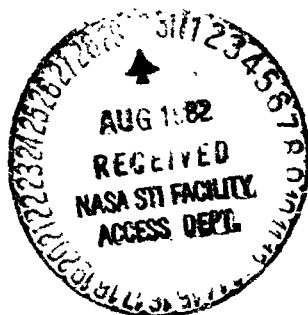# Extraterrestrial Materials Processing

Wolfgang H. Steurer

April 15, 1982

NASA

National Aeronautics and
Space Administration

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

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PREFACE

The work documented in this report was sponsored by the Materials
Processing Office of OSSA and carried out jointly by JPL's Materials Research
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of this report.

Figure IV - 6 was reprinted with permission from F.H. Norton, Elements of Ceramics, Addison-Wesley Press Inc., 1952.

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EXECUTIVE SUMMARY

This report summarizes the results of the first year of a multiyear study on the processing of extraterrestrial materials for use in space.

The basic physics of launch and transport energies indicate that, at least theoretically, significant advantages can be derived from the large-scale use of extraterrestrial materials; an objective of this study is to generate the information which will ultimately permit evaluation of the potential for practical achievement of these advantages.

The initial part of the FY'81 effort was devoted to the necessary accumulation, sifting and analysis of a large body of relevant in .rmation on extraterrestrial resources and terrestrial processes and assessment of a broad spectrum of potential extraterrestrial process and product concepts. This background was essential to reach an appropriate focus at approximately mid-year on those issues requiring detailed analysis and experimental investigations and establishment of the basis for programmatic priorities.

Candidate product/applications include construction materials for earth-orbital or lunar surface systems, materials for radiation shielding, propellants for transport vehicles, semiconductors for solar power systems and volatiles for life support. Construction materials fall into two general categories: (1) products similar to those common to terrestrial and conventional space construction (e.g., shaped metal alloy parts) which require complex processes and facilities; and (2) products adapted to space resources (e.g., mixed silicate crystalline or glassy components) which require relatively simple processes but call for new concepts or re-examination of old concepts of design and construction.

Available raw materials include: silicate-rich mixed oxides on the Moon, some asteroids and Mars; free metals, primarily iron and nickel in some asteroids and in small quantities in the lunar soil; volatiles like water and CO_2 on some asteroids and Mars. There is no evidence of ore bodies (concentrations of particular minerals) on the Moon but the lunar soil contains useful amounts--as oxides--of Fe, Al, Si, Ti, Ca and Mg. The general similarity between known or postulated extraterrestrial materials and certain terrestrial minerals allows formulation of mock extraterrestrial materials for use in process experiments.

Operations for raw material acquisition (mining) and conditioning have been examined. The type and extent of such operations will be strongly dependent on the raw material resource and the required output feedstock for subsequent processes. They will likely include both those derived from terrestrial operations, but adapted to the extraterrestrial environment (e.g., grinding, sizing), and those specifically designed to utilize the extraterrestrial environment (e.g., in-situ melting of lunar surface material).

Processes for extraterrestrial materials/products are likely to be significantly different than their terrestrial counterparts largely because of major differences in the environment. The differences (except for Mars) are 1) absence of atmosphere; 2) lack of readily accessible working fluids (air, oil, water); 3) low gravity ranging from 0.2g on the Moon to 10^{-5}g on smaller asteroids; 4) solar energy not attenuated by atmospheric absorption or scattering and 5) severe constraints on the degree and frequency of manned intervention.

Six categories of primary processes have been identified:

A.   Silicate products from silicates.
B.   Metals and oxygen from silicates.
C.   Metal products from free metals.
D.   Hydrogen from lunar soil.
E.   Propellants from bound or free water or hydrogen.
F.   Carbon from carbonaceous chondrites.

Primary efforts are concentrated on the first two. Preliminary studies are being carried out on C and D. Categories E and F will be deferred since

free water on the Moon in the form of polar permafrost is considered unlikely and extraction of H_2O and carbon from asteroids is a long range objective.

Analytical studies of silicate products and initial experiments with synthetic extraterrestrial raw materials have been completed, defining the effective ranges of processing conditions and temperatures. Evaluation of the experimental results indicates that both polycrystalline and amorphous products can be produced, depending on specific starting materials and processing conditions.

With regard to the extraction of metals and oxygen from extraterrestrial raw materials, many of the chemical processes which have been suggested appear to be impractical because of equipment complexity and the requirement for large quantities of processing agents which have to be imported from earth. In contrast, the extraction of metals and oxygen by magma electrolysis and by vapor/ion phase separation appears to be both practical and cost efficient.

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ABSTRACT

This report summarizes the first year results of a multi-year study of
processing extraterrestrial materials for use in space. Theoretically, there
are potential major advantages to be derived from the use of such materials
for future space endeavors.

This report describes the types of known or postulated starting raw
materials including silicate-rich mixed oxides on the Moon, some asteroids and
Mars; free metals in some asteroids and in small quantitites in the lunar
soil; and probably volatiles like water and CO_2 on Mars and some asteroids.

Candidate processes for space materials are likely to be significantly
different from their terrestrial counterparts largely because of: absence of
atmosphere; lack of readily available working fluids; low- or micro-gravity;
no carbon-based fuels; readily available solar energy; and severe constraints
on manned intervention. The extraction of metals and oxygen from lunar
material by magma electrolysis or by vapor/ion phase separation appears
practical.

Section I

OBJECTIVES AND SCOPE

This is the first annual report on RTOP 179-29-62-01 entitled "Extraterrestrial Materials Processing." Its purpose is to outline the approaches which have been chosen and to give an account of the work which has been carried out at JPL during the first year of the program.

This program is based on the rationale that the energy required to move material from the earth's surface, to low earth orbit or beyond, is significantly greater than to move an equivalent mass from the Moon, Mars or the asteroids to the same place in space. Therefore, there is the potential for a significant advantage to be gained from the large scale use of extraterrestrial materials for space equipment and operations. There is inadequate information on candidate processes and products to determine if the theoretically possible advantages can be practically achieved.

The referenced RTOP called for a 3-year study and experimental program on processes for the recovery of materials from extraterrestrial sources, primarily the moon and near-earth asteroids, for use in the construction of advanced space systems and for re-fueling of space transport vehicles. The specific objective of the 3-year program was to define, develop and experimentally evaluate appropriate processes for the extraction of useful materials from typical extraterrestrial minerals adapted to, and utilizing the prevailing environmental resources and conditions. The original RTOP further called for the study of potential process automation, starting in the second year.

The RTOP has been modified for FY'82 and beyond as requested by NASA. The new plan calls for a five year research program with the overall

objectives of developing a technology base relevant to acquisition and processing of extraterrestrial materials that will: 1) enable future examination of the practicality of such processing and 2) be the foundation of scientific understanding on which an effective process development program can be built when such a program is initiated.

The first year (FY'81) effort was devoted to (1) the survey of extraterrestrial raw material resources and (2) the definition and evaluation of several promising processing concepts by means of theoretical and technological studies, supported by initial exploratory laboratory experiments.

This report represents the results of the FY'81 effort. In accordance with the program plan, it documents the theoretical and technological investigations which were the essential basis for experiment planning, as well as the results of the initial experimental investigations.

Section II
STUDY APPROACH

A. BASIC CONSIDERATIONS

The subject of this program is based on the premise that the utilization of extraterrestrial materials in the construction of space systems promises substantial cost savings in space operations, primarily due to the elimination of the costly launch of materials from earth. Numerous papers have been written on this subject (Ref. 1-42). Even though most of these studies assess the technological and cost elements of space operations in great depth, they are necessarily based on assumptions as to the cost of extraterrestrial materials production and the related support operations. Any prediction of economical gains is, therefore, highly speculative and questionable as long as no hard data on extraterrestrial production cost are available.

The present study is designed to generate more reliable data on the efforts required to produce materials from extraterrestrial minerals and on the related cost. At the same time the study will provide the foundation for the development of effective extraterrestrial processes and the requisite production facilities.

With regard to economics, the most important aspect of process effectiveness is the need to supply materials and provide support from earth. The major imports are likely to be: 1) processing equipment, 2) power plant, 3) consumables, 4) propellants for product transport and 5) manned operational support.

In the foreseeable future, processing equipment will be built on earth and transported to point of use. It represents a one-time investment which can be written off over the projected years of operation or, more accurately, for the tons of material produced over the equipment lifetime. Eventually, it is possible that an initial facility can be developed and installed that can produce components which will expand and/or reproduce the original, reducing the supply of equipment required from earth for expanded production.

For most of the candidate processes, significant quantities of thermal and/or electrical energy will be required. The facility to convert nuclear or solar energy into the appropriate form is likely to represent a major import from earth. It is possible that portions of the power plant (e.g., solar concentrators and radiation shielding) can be produced from extraterrestrial materials, reducing required imports.

Consumables such as chemicals or components subject to wear may prove to be more significant than processing or power equipment since they contribute to the import cost of every ton of material produced. The demand for consumables will be a major criterion for process viability.

Consumption of earth-origin propellant to transport extraterrestrial products to their point of use will detract from the advantages of these products' use in proportion to the amount of earth origin propellant required. Production of some or all of the propellant from extraterrestrial sources is possible. However, process equipment and power supply to produce such extraterrestrial propellant must be charged against the ultimate product delivered.

Manned operational support will probably be required, particularly for operation start-up and for non-routine repairs/maintenance. Routine operations and maintenance will be automated and/or carried out by teleoperated robots. Excessive manned support could quickly eliminate potential advantages of using extraterrestrial materials.

8. PRODUCTS AND PRODUCT APPLICATIONS

Another basic consideration is the type of products to be produced for use in space systems and missions. There are two alternatives: (1) Products

as they are commonly used in terrestrial systems design and construction, such as single metals or alloys (extraterrestrial production of polymer-base materials does not appear feasible for the foreseeable future due to the lack of carbon and hydrogen): these metal products require complex processes and space facilities, yet need little change in systems design. (2) Products which can be obtained directly from the indigenous raw materials, such as silicate or glass components. They require substantially less processing efforts, essentially for conversion into the product shape and/or for the improvement of properties, yet necessitate the adoption of new concepts of systems design and in-space construction.

MATERIAL TYPES	MASS	STRENGTH	DESIGN	COST
	LIGHT	HIGH	CONVEN- TIONAL	HIGH
METAL ALLOYS				
PURE METALS				
METAL MIXTURES				
COMPOSITES				
PARTICLE COMPACTS				
CAST BASALT				
	MASSIVE	LOW	NEW DESIGN CONCEPTS	LOW
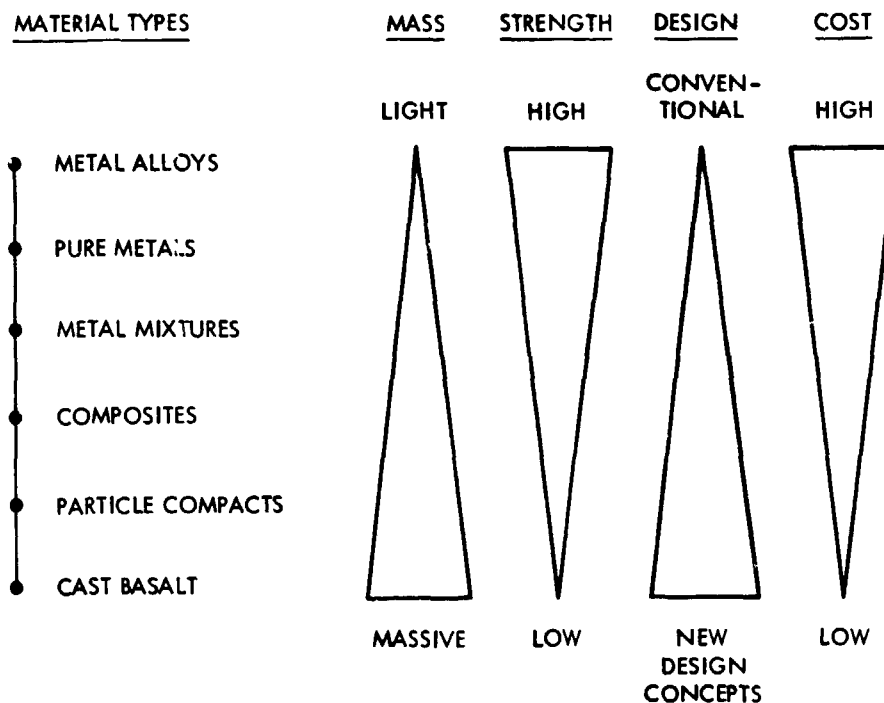
Figure II-1 Product Choice: Design and Cost Implications

There are, of course intermediate choices between these two extremes, such as the use of products from unrefined metal mixtures which can be produced at substantially less effort and cost than specific metals or alloys. A number of product choices are listed in Figure II-1, together with a qualitative indication of their relationship to component mass and strength, systems design and production cost. At present, no choice of products has been made in this study and the entire spectrum of potential materials is being considered in the evaluation of processes.

The qualitative assessment of expected characteristics of Figure II-1 is typical of the present project approach to product and process assessment in that available information does not warrant introduction of quantitative data. Likewise, systems requirements are assessed in terms of primary functional characteristics, such as thermal stability or stiffness, rather than using properties which depend entirely on specific designs.

C.   RAW MATERIAL RESOURCES

¯he extraction of useful materials and products from extraterrestrial mineral resources comprises the following major topics:

(1) The definition of extraterrestrial resources in terms of minerals and environments.

(2) The acquisition ("mining") of raw materials and their conditioning for subsequent processing.

(3) The processing of conditioned raw materials into useful products.

In the definition of available raw materials, prime emphasis is placed on the moon and the asteroids, particularly those whose orbits approach or cross the orbit of the Earth. The main belt asteroids and the Mars system are included in the definition, but not further pursued with regard to processing at this time in view of their more difficult accessibility.

The subject of material resources is being addressed in two ways: (1) by a study of the state of knowledge on planetary mineralogy, and (2) by extracting from this study a definition of raw materials adaptable to processing into products, identifying such technological characteristics as composition, raw material form and location.

In view of the basic importance of the knowledge of the availability and nature of extraterrestrial raw materials, assistance was sought and obtained from experts in planetary sciences and meteoritics. An interface was also established with the ongoing development of asteroidal exploration missions.

The efforts on raw materials further included the preparation of so-called "mock materials" (synthetic extraterrestrial mineral combinations derived from terrestrial minerals) for use in processing experiments.

D. RAW MATERIAL ACQUISITION AND CONDITIONING

The work on raw material acquisition, the equivalent of terrestrial mining, consisted exclusively in the conceptual development of space-adapted techniques. The peculiar environmental conditions and the operational constraints encountered in space suggest a departure from conventional techniques. An example of such departure is acquisition by in-situ melting of the surface material using direct solar energy.

The studies on raw material conditioning leaned extensively on work performed and reported by other investigators, part ularly by R. Williams et al. at JSC (Ref. 68).

E. PROCESSING

The evaluation and development of extraterrestrial processing methods represents the primary effort of this study. It was divided into three tasks:

Task 1   Selection of processes for detailed study.

Task 2   Theoretical and technological evaluation of the selected processes.

Task 3   Experimental investigations.

The first step in Task 1 was a survey of terrestrial processes and proposed space processes (Ref. 27-29, 34-39, 64-72, 82, 89) as well as the identification of new processing concepts. The processes were examined in the context of adaptability to extraterrestrial conditions and operations such as raw material resources, import requirements, utilization of environmental resources, process simplicity/complexity, expected product yield and quality, facility requirements as well as operational support requirements. Those processes characterized by the highest adaptability to space conditions were selected for further study in Task 2 and Task 3, while the remaining processes were either eliminated or temporarily shelved. The selected processes were: (a) solid-state processing of silicates into silicate products; (b) liquid-state processing of silicates into silicate or glass products; (c) production of metals and oxygen by electrolysis of molten silicates and (d) production of metals and oxygen by vapor-phase reduction.

In Task 2 the selected processes are being evaluated in depth in theoretical and technological studies, including conceptual facility design and preliminary assessment of operational support requirements. One of the most significant objectives of this task is the definition of those material characteristics and property interactions which play a significant role in process effectiveness, either in terms of yield or product quality. Attempts are being made to generate the pertinent data by literature survey or theoretical studies. Wherever this is unsuccessful, appropriate laboratory experiments are designed and carried out to provide the necessary information.

The experimental investigations (Task 3) comprise (1) exploratory experiments with various minerals to gain an understanding of their behavior under simulated extraterrestrial processing conditions and for go/no-go decisions, (2) establishment of generally applicable data on the basic properties of typical raw materials, in the solid, liquid and gaseous state and (3) experimental investigation of the critical material characteristics for specific processes identified in Task 2 and generation of pertinent property data. In all cases simulated raw materials, synthesized from terrestrial minerals, are used for experimentation. The gradual build-up of experimental facilities and experience is oriented toward the development of the capability to perform complete process demonstration experiments.

The investigations carried out to-date are documented in the subsequent chapters as follows:

Section III: Extraterrestrial Raw Materials
Section IV: Mining and Material Conditioning
Section V: Processing - Basic Considerations
Section VI: Processes

Some uncommon terms, used in this report, are defined in Appendix A. The sources of asteroid information are discussed in Appendix B.

Section III

EXTRATERRESTRIAL RAW MATERIALS

A. OVERVIEW OF RAW MATERIAL RESOURCES

The raw materials available in space can be simply described as composed
of a mixture of silicates, other oxides, and in some cases metals. The
silicates usually contain various combinations of iron, magnesium, calcium,
sodium and aluminum. Eighty-nine specific minerals have been identified among
the meteorites alone and others probably exist. Some extraterrestrial
minerals were previously unknown on Earth while others are common terrestrial
minerals. The fine scale structure can vary from dust, to loosely aggregated
masses, to hard interlocking crystals. In all cases there is more to learn
about the geology and availability of extraterrestrial mineral resources.

In the first approach, extraterrestrial materials may be classified
according to the richness in metal, volatile, and silicate components as shown
in Figure III-1. Permafrost represents the most concentrated form of volatile
and would be expected on Mars, and possibly also at the lunar poles and on
cometary bodies. Carbonaceous chondrites* also have appreciable H_2O (0-20%)
present. In contrast, the lunar rocks and achondritic material in asteroids,*
are volatile-poor and relatively rich in silicate material. Mars may also
have silicate deposits present. The Ni-Fe metal which may be found in some
asteroid* bodies is both silicate-and volatile-poor.

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* As discussed in Section III-B-2 and Appendix B, composition, morphology
and crystallography of asteroids are inferred from examination of
meteorites and from remote observations.

Figure III-1   Classification of Extraterrestrial Raw Materials
According to Volatile, Silicate and Metal Content

The mineralogic assemblages expected at different sources are identified in more detail in Table III-1. Mars contains the greatest variety of materials available. Special materials, such as Ni-Fe (asteroidal) or ilmenite (a potential lunar ore of titanium) can be found elsewhere. The lunar resources, however, lack volatiles such as water unless present as permafrost in the lunar pole craters, and the asteroidal resources may lack volatiles, silicates, or metals, depending on the particular asteroid considered.

For the purpose of mining and subsequent processing into useful products, the most important characteristics of extraterrestrial raw material e composition, form and location. The following three tables are de   d to convey an overview of these characteristics. (For more detailed di   ion see subsequent Section III-B.) They comprise the moon and the predominant types of asteroids (ordinary chondrites, carbonaceous chondrites, stony irons and irons) in view of the pronounced difference in their mineralogical characteristics. These tables were assembled from in-depth analysis of the information contained in References 43-47 (Moon) and 48-55 (asteroids). It is emphasized again that most of the asteroid data are inferred from meteorites, since astronomical observation permits only a first-order classification.

With regard to composition, the defined sources represent a wide variety of minerals from all-silicates to all-metals, as illustrated in Table III-2. It can be seen that the Moon is composed almost totally of silicates (metal oxides), with only trace amounts of free metals and hydrogen. This applies at least to the crust in the equatorial region; the potential presence of water in the form of polar permafrost is omitted, as it is not verified and is questionable. The Ni-Fe asteroids ("Irons") on the other hand are characterized by essentially 100% free metals and a nearly complete absence of volatiles. The ordinary chondrites exhibit appreciable amounts of free metals, while the carbonaceous chondrites have virtually no free metals, yet contain carbon and water. Both chondrite types are of particular interest as they probably represent the bulk of the more accessible earth-approaching asteroids.

Table III-1   OVERVIEW OF EXTRATERRESTRIAL MINERALS

LUNAR

Pyroxene (augite and pigeonite)   Fe,Mg Ca silicate
Plagioclase (anorthite)   Ca, Al silicate
Olivine   Fe, Mg silicate
Ilmenite   Fe, Ti oxide

ASTEROIDAL*

Pyroxene (hypersthene, bronzite, enstatite)   Fe, Mg silicate
Plagioclase (oligoclase, anorthite)   Na, Ca Al silicate
Olivine   Fe, Mg silicate
Troilite   Fe Sulfide
Layer Lattice silicates   Complex minerals with
                                                Mg,Si,OH,Al,O,Ca,Na
Magnetite   Fe oxide
Nickel-Iron   NiFe alloy

Various types of asteroids exhibit different combinations of these minerals:

Irons: Nickel-Iron, Troilite, small amounts of graphite
Ordinary Chondrites: Olivine, Pyroxene, Troilite, Plagioclase,
                           Nickel-Iron
Carbonaceous Chondrites: Layer Lattice Silicates, Magnetite, Olivine,
                           Troilite, Pyroxene
Stony Irons: Nickel-Iron, Troilite, Olivine and/or Pyroxene

MARTIAN

Surface:

   Probable basalt consisting of pyroxene, olivine and plagioclase
   Surface fines of iron rich clays and iron oxide with various contaminants
   Probable evaporite deposits (salts and carbonates)
   Water and CO_2 ice in certain locations

Moons:

   Probably brecciated carbonaceous chondrite material.

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*   As discussed in Section III-B-2 and Appendix B, composition, morphology
     and crystallography of asteroids are inferred from examination of
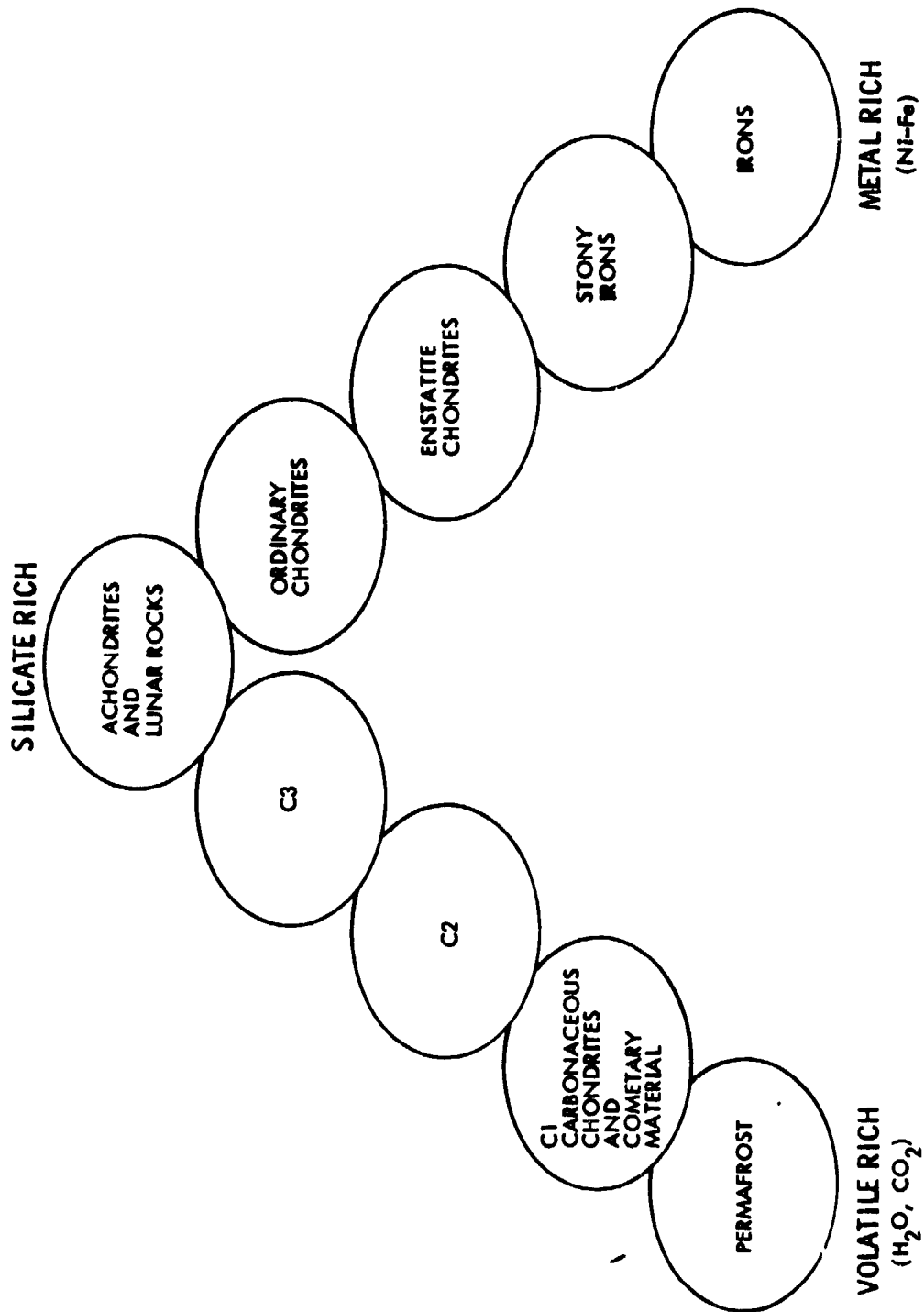   meteorites and from remote observations.

Table III-2 <u>TYPICAL COMPOSITION OF THE MOON AND ASTEROIDS</u>

(%)

OBJECTS	METAL OXIDES	FREE METALS	FeS	C	H_2O	H_2
MOON	99	<1	-	-	-(xx)	0.01 (x)
ORDINARY CHONDRITES	85	8.3	6	-	-	-
CARBONACEOUS CHONDR.	75	<1	8.6	2.4	~13	-
STONY IRONS	43	53	3	-	<1	-
IRONS (Fe-Ni-Co)	-	98	1.8	-	-	-

(x) IN SOIL TO DEPTH OF ~1m ONLY

(xx) POTENTIALLY (TBD) AS PERMAFROST IN POLAR REGIONS ONLY

The composition of lunar and asteroidal raw materials is defined in detail in <u>Table III-3</u>. This chart identifies composition ranges, based on the present state of knowledge, as well as "point compositions" (analogous to "point designs") chosen by best judgement. The point compositions serve as raw material baseline for this study.

The form and location of the raw materials, as well as their potential usefulness with regard to processing into products, are illustrated in <u>Table III-4</u>. Of foremost interest with regard to mining and conditioning are the various raw material forms, such as hard rocks, friable rocks or soil (regolith).

B. MINERALOGY OF EXTRATERRESTRIAL BODIES

The bodies that have been selected for this study as sources of raw materials are the Moon, the asteroids, and the Martian system (planet and its moons). Each presents a unique environment, and therefore composition and morphology.

Table III-3

COMPOSITION OF LUNAR AND ASTEROIDAL RAW MATERIALS

SEE NOTES (1) (2) BELOW	LUNAR ANORTHITE		LUNAR BASALT		ORDINARY CHONDRITES		CARBONACEOUS CHONDRITES		STONY IRONS		IRONS	
	POINT	RANGE	POINT	RANGE	POINT	RANGE	POINT	RANGE	POINT	RANGE	POINT	RANGE
SiO_2	45.5	44 - 45	38	37 - 41	39.7	36 - 40	28	23 - 33	21	17 - 35	—	—
MgO	6.3	6 - 7.6	9.9	6.8 - 10	24.6	23 - 25	20	15 - 23	7.	6.4 - 23	—	—
FeO/Fe_2O_3	5.6	5.1 - 6.2	18.6	18.2 - 19.6	14.3	9 - 14.3	20	10 - 24	7.5	4.4 - 7.7	—	—
Al_2O_3	26.4	26 - 27	8.9	6.8 - 10	2.8	2.6 - 2.8	2.3	1.7 - 2.7	4.0	0 - 4.1	—	—
TiO_2	0.27	0.2 - 0.4	13.0	10 - 13	0.11	0.11	0.10	0.08 - 0.12	—	—	—	—
CaO	15.2	15.1 - 15.4	10.1	10 - 12	1.9	1.87 - 1.92	2.0	1.5 - 2.3	2.8	0 - 3.0	—	—
Na_2O	0.33	0.3 - 0.35	0.35	0.32 - 0.44	0.94	0.93 - 0.94	0.54	0.54 - 0.76	0.16	0 - 0.17	—	—
K_2O	0.08	0.06 - 0.08	0.06	0.04 - 0.09	0.11	0.10 - 0.11	0.05	0.05 - 0.07	—	0 - 0.03	—	—
MnO	0.1	0.06 - 0.1	0.28	0.28 - 0.29	0.26	0.26	0.17	0.17 - 0.20	0.20	0 - 0.22	—	—
Cr_2O_3	0.14	0.11 - 0.14	0.61	0.27 - 0.61	0.41	0.33 - 0.41	0.39	0.28 - 0.51	0.36	0 - 0.68	—	—
NiO	—	—	—	—	—	—	1.56	0.3 - 1.56	0.40	0 - 0.4	—	—
TOTAL METAL OXIDES	99.9		99.8		85.1		75.1		43.4		– 0 –	
Fe	—	—	—	—	7.13	7.1 - 17.4	—	0 - 2.3	48	28 - 49	88.9	80 - 93
Ni	—	—	—	—	1.07	1 - 1.7	0.16	0 - 1	4.4	4 - 4.7	8.1	5.5 - 19
Co	—	—	—	—	0.07	0.07 - 0.1	—	0 - 0.06	0.28	0 - 0.3	0.58	0.5 - 1.0
Pt/Au	—	—	—	—	—	—	—	—	?	?	26 ppm	4 - 49 ppm
TOTAL FREE METALS	– 0 –		– 0 –		8.3		0.16		52.7		97.6	
FeS	—	—	—	—	6.06	5.6 - 6	8.6	6 - 17	2.8	0 - 7.4	1.8	0.6 - 4.6
C	—	—	—	—	—	—	2.4	0.5 - 3.6	—	0 - 0.08	0.06	0.01 - 0.6
P_2O_5/P	0.04	0.03 - 0.05	0.06	0.05 - 0.09	0.26	0.23 - 0.26	0.27	0.27 - 0.32	0.11	0 - 0.11	0.08	0.15 - 0.2
S	0.04	0.03 - 0.15	0.15	0.15 - 0.19	—	—	—	TRACE	—	—	0.18	—
H_2O	—	—	—	—	0.24	0.24 - 0.30	13.2	1 - 20.5	0.7	0 - 0.7	—	—

(1) ALL DATA IN % BY MASS, EXCEPT WHERE INDICATED OTHERWISE

(2) "POINT" COMPOSITIONS ARE NOMINAL VALUES BASED ON BEST AVAILABLE EVIDENCE AND JUDGEMENT. THEY SERVE AS STUDY BASELINE

TABLE III-4 FORM AND LOCATION OF RESOURCES

RAW MATERIALS	LOCATION			SOLID PRODUCTS		VOLATILES				C
	MOON	ASTE-ROIDS	MARS SYST.	METALS	NON-METALS	O_2	H_2	H_2O	CO_2	
HARD SILICATE ROCKS	●	●	●	●	●	●				
CARBONACEOUS ROCKS		●	(?)	●	●	●	←--●	●	●	●
SILICATE + METAL ROCKS		●	(?)	●	O	O				
ALL METAL BODIES		●		●						
SILICATE SOIL	●	O	O	●	●	●--O--▷				
CARBONACEOUS SOIL		●	(?)	O	●	●	←--●	●	●	●
PERMAFROST	(?)	(?)	●			←-	←-	●	O	

● APPLICABLE LOCATIONS AND PRODUCTS   (O POTENTIALLY APPLICABLE)

●- ➜ SECONDARY PRODUCT EXTRACTION

1. The Moon

There are two main geologic provinces on the Moon's surface: The
lunar highlands and the lunar maria. In both cases the indigenous material is
made up of various fragment sizes ranging from fine dust to large rocks, and
composed of mineral fragments, rock and glass fragments and breccia (welded
rubble) fragments. The lunar highlands are characterized by the presence of
anorthosite which is a rock with large amounts of calcic plagioclase, and
minor amounts of a mineral named KREEP which is a silicate rich in potassium,
rare earth elements and phosphorous. The lunar maria are characterized by the
presence of basalt which is rich in iron and magnesium silicates and which may
have a crystalline texture. The lunar basalt is unusual by earth standards in
that it may contain a large amount of ilmenite but no magnetite (it was formed
under highly reduced conditions). The basalts in the maria vary somewhat in
chemical composition. There have been rocks of other composition (dunite) and
colored glasses found at the Apollo landing sites so the geology should be

considered more complex than as described here. Thanks to the Apollo program we are assured that certain raw materials can be found at the Apollo sites although additional rocks may also exist. There is no evidence for lunar ore concentrations or other bodies of mineral enrichment, like those found on earth. (The lunar MASCONS may be concentrations but are probably too deep to be practical materials sources.)

Table III-5 <u>ABUNDANCE AND PROPERTIES OF THE PREDOMINANT LUNAR MINERALS</u>

| MINERAL | COMPOSITION | ABUNDANCE % | | | | DENSITY g/cc | MELTING TEMP °C | STRENGTH (RT) bars |
		BASALT	ANORTH	BRECCIA	SOIL			
PYROXENE	$MgSiO_3$ $CaSiO_3$ $FeSiO_3$	40-65	0-40	5-30	5-20	2.8-3.7	1550	17,000
OLIVINE	Mg_2SiO_4 Fe_2SiO_4	0-35	0-40	1-5	0-4	3.20 4.39	1890 1205	21,000
PLAGIO-CLASE	$CaAl_2Si_2O_8$	15-35	40-98	15-90	10-60	2.734	1550	6,000
ILMENITE	$FeTiO_3$	0-25	0	1-12	1-5	4.7	1400	

(SOURCE: R. WILLIAMS, HANDBOOK OF LUNAR MATERIALS (Ref. 43))

The rocks of the Moon (Ref. 43-47) are dominated by four minerals: pyroxenes, olivine, plagioclase feldspars and ilmenite. The abundance and major characteristics of these minerals are summarized in <u>Table III-5</u>. There are many other minerals reported although in smaller quantities. The pyroxenes can be chemically described as a mixture of $MgSiO_3$, $CaSiO_3$ and $FeSiO_3$. They form two minerals: augite and pigeonite. Pigeonite is distinguished from augite in that it has more Ca present. Alumina (up to 12% Al_2O_3), titanium (up to 5% TiO_2), and chromite (up to 1.25% Cr_2O_3), are accepted into the mineral in solid solution. The mare basalts contain 40 to 65 percent pyroxene which is mostly augite. The pyroxene in the anorthosites is 0 to 40 percent and mostly pigeonite. Olivine is a mixture of

Fe_2SiO_4 and Mg_2SiO_4 with the ratio generally between 50 and 75 percent of the Mg rich end member. The olivine is volumetrically zero to a few percent in most minerals and up to 35% in the mare basalts, although dunite (rock of nearly pure olivine) has been found. The feldspar is a solid solution of $CaAl_2Si_2O_8$ and $NaAlSi_3O_8$ with the Ca rich end member usually greater than 90% by mole fraction. The plagioclase fraction of the lunar rocks varies from 15% in the mare basalts, to 90% in the anorthosites. The ilmenite is $FeTiO_3$ with small amounts of $MgTiO_3$. The ilmenite abundance is usually less than 2% but in some mare basalts may reach over 10%.

The structure of the material can be rock (basalt and anorthosite), broken and welded fragments of rock (breccia) or finely broken fragments of rock and glass which are not welded together (soil). The composition of the soil is grossly similar to the composition of the underlying rock. Thus in a Ti-rich basalt the soil will also be Ti-rich. The particle size distribution of the soil, as derived from Ref. 43, is defined in Figure III-2. Most soils are produced by meteorite impact and thus contain glass. The glass often bonds mineral fragments together producing "agglutinates". Breccias may contain up to 50% glass. Some fine grained anomalous soils (such as the black and orange soils collected at the Apollo 17 site) are pyroclastic ejecta and are finer grained than most soils.

The mineralogical composition of the predominant lunar raw materials may be summarized as follows:

Lunar Anorthosite (Average Mineralogy, weight percent) (Ref. 43)

plagioclase (Ca rich)	83%
olivine	16%
pyroxene (primarily pigeonite)	1%
ilmenite	1%

Lunar Mare Basalt (Average Mineralogy, weight percent) (Ref. 44)

Pyroxene (primarily augite)	50%
plagioclase (Ca rich)	27%
ilmenite	20%
olivine	3%
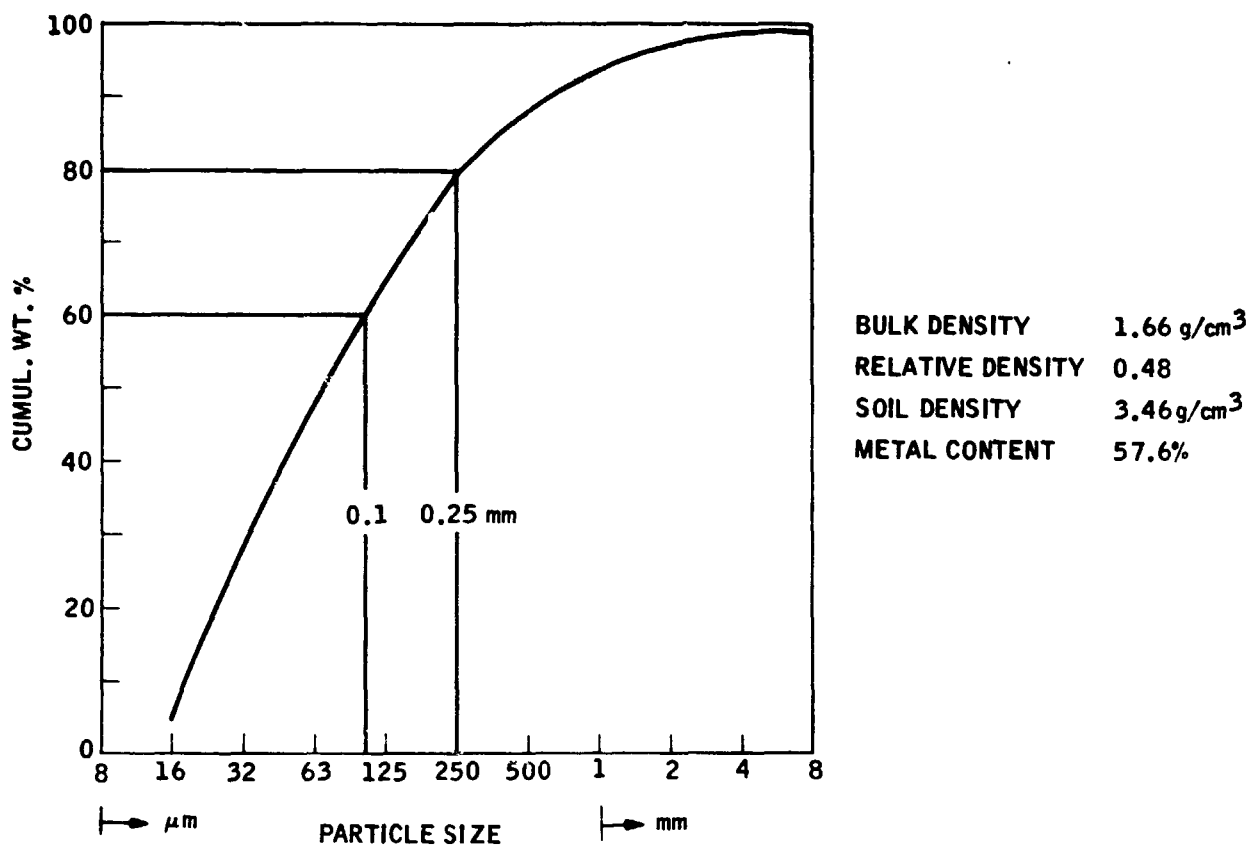
Figure III-2   Particle Size Distribution of the Lunar Soil
(derived from Ref. 43)

2.   The Asteroids

Asteroids and comets are bodies that constitute solid, rocky matter in the solar system which is larger than dust and smaller than planets. Most asteroids are between the orbits of Mars and Jupiter (main belt), but some are found within the orbits of the terrestrial planets. Some asteroids have orbits which cross the orbit of the Earth. These are called "Earth crossers" or "Apollo" objects. The moons of Mars are also probably asteroid-like bodies (Ref. 56, 57).

The size of the earth approaching objects ranges from small fragments to tens of kilometers (across largest dimension). So far some 40 objects of more than 700 m across have been identified and defined with regard to orbital characteristics and mineral type. However, many thousands of smaller

asteroids are expected to exist (Ref. 58, 59). (Even under most favorable conditions as to proximity and albedo, objects of less than 300 m are below the sensitivity of today's telescopes (Ref. 60).) The largest one known today is Eros (~20 km). To place these sizes in perspective, one should realize that a 700 m object of the ordinary chondrite type represents a mass of approximately 500 million tons, a 2 km object of 12 billion tons and a 10 km object 1.5 trillion tons.

Some of these objects are close to earth relative to other planetary bodies. A rendezvous mission to some of these objects, such as Anteros (1.3 km), can be accomplished with less launch energy than required for Moon missions (Ref. 61). However, the orbital periods in the order of 1.5 years limit the opportunities for low delta Δv missions given the presently known asteroids.

The spin rates of asteroids vary from around 0.5 to 6 (usually 2.5 to 3) rotations per Earth day. Large asteroids tend to spin more slowly than small ones, and Earth-crossing asteroids often have high spin rates. Also very large "C"-type asteroids spin faster than most. Occultation data suggest that swarms of smaller bodies may orbit some asteroids but the results are still unreliable. The asteroids appear to be of various shapes, from roughly spherical to elongated (Ref. 48).

The earth is constantly rained upon by considerable debris from space which may be related to asteroids and comets. Most of the geochemical information about the asteroids comes from studies of meteorites and extraterrestrial dust and the assumption that meteorites are representative of asteroids. A substantial fraction of the debris burns up in the atmosphere. Some of the stronger, slower and larger bodies are able to withstand the atmospheric passage and impact the surface. Once on Earth, they are designated meteorites. Analysis of meteorites, supported by spectral studies of asteroids in space and meteors (the streak of a "falling star"), provides the basis for implied composition and morphology of the asteroids. There is general similarity between the meteorite mineralogy and inferred mineralogy from telescopic examination of asteroids (Ref. 49-53), but only a few meteorites have been reliably associated as mineralogically very similar to an asteroid. This may be because the asteroids are small, and often have low albedoes, although it is possible that the compositions of asteroids are

different from meteorites. Spectrally, the prevalence of carbonaceous-like material is much greater than of the ordinary chondrite-like material. Stony material has been associated with Earth-crossers. Carbonaceous chondrites have been associated with the majority of main belt asteroids, although the carbonaceous bodies are found in earth-crossing orbits as well. All bodies over 200 km in diameter probably have surfaces with carbonaceous chondrite or basaltic compositions (Ref. 54).

The meteorites can be described as composed of three components: volatile, silicate and Ni-Fe metal (see Figure III-1). There are no meteorites with both high volatile content and high metal content. There are four basic types of meteorites and thus rock types representative of asteroidal bodies (more rock types may exist which have not yet been discovered):

(1) Chondrites - composed mostly of a fine grained silicate matrix with or without small spherules of glass or high temperature mineral (which are called chondrules).

(2) Achondrites - composed of interlocking crystals of silicate as if solidified from a silicate melt.

(3) Irons - composed of nearly all Ni-Fe alloy.

(4) Stony-Irons - composed of a mixture (about 50/50) of silicates and Ne-Fe alloy.

The most common minerals found in meteorites and thus presumably in asteroids are:

olivine	$(Mg, Fe)_2 SiO_4$
pyroxene	$(Mg, Fe, Ca) SiO_3$
troilite	FeS
magnetite	Fe_3O_4
layer lattice silicates	a complex mixture of Mg, Si, OH, Al, O, NA
nickel-iron	Ni, Fe
plagioclase	$NaAlSi_3O_8 - CaAl_2Si_2O_8$

There are many other minerals found in meteorites which are apparently less abundant.

The range of oxide and met..l compositions potentially prevailing among asteroids - as inferred from meteorite compositions - is shown graphically in Figure III-3 (Ref. 53, 55). Various systematic variations are apparent in the amounts of various oxides present. Ni-Fe metal can vary over a wide range, while SiO_2 can only vary from 0 to around 50%. CaO does not appear to rise above 30%. Of course, there may be other mineral assemblages and oxide compositions in space, but the meteorites can at least provide a general idea as to what oxide compositions are likely.

The chondrites are the most common type of mineral assemblage in near-earth space. Carbonaceous chondrites are probably the most common asteroidal rock type in near-Earth space. (For reasons see Appendix B.) The next most common type is probably the ordinary chondrite (also known as Bronzite and Hypersthene chondrites). Nickel-iron rich bodies rarely encounter the earth as compared to the other basic types. The reason why they are so common in meteorite collections is because they are resistant to the stresses of atmospheric passage and are peculiar looking as compared to normal terrestrial rocks.

The mineralogical compositions and structures of the various types of meteorites are described in Appendix B.

3. Mars

Mars is a planet which is probably dominated by volcanic activity, widespread well-mixed dust deposits, massive water and carbon dioxide ice deposits, and saline deposits. The most geologically similar places on Earth are the dry valleys of Antarctica. In these perennially frozen valleys, as on Mars, there is salt cementation of sediments and extremely saline pockets of groundwater. On Mars the composition of the major bedrock types is, as on the Moon, probably basalt. The environment is not so reducing due to the presence of abundant volatiles, however, and different mineral assemblages should be expected. The virtually omnipresent olivine, plagioclase, and pyroxene will probably be present on Mars as well. The fine grained materials sampled by
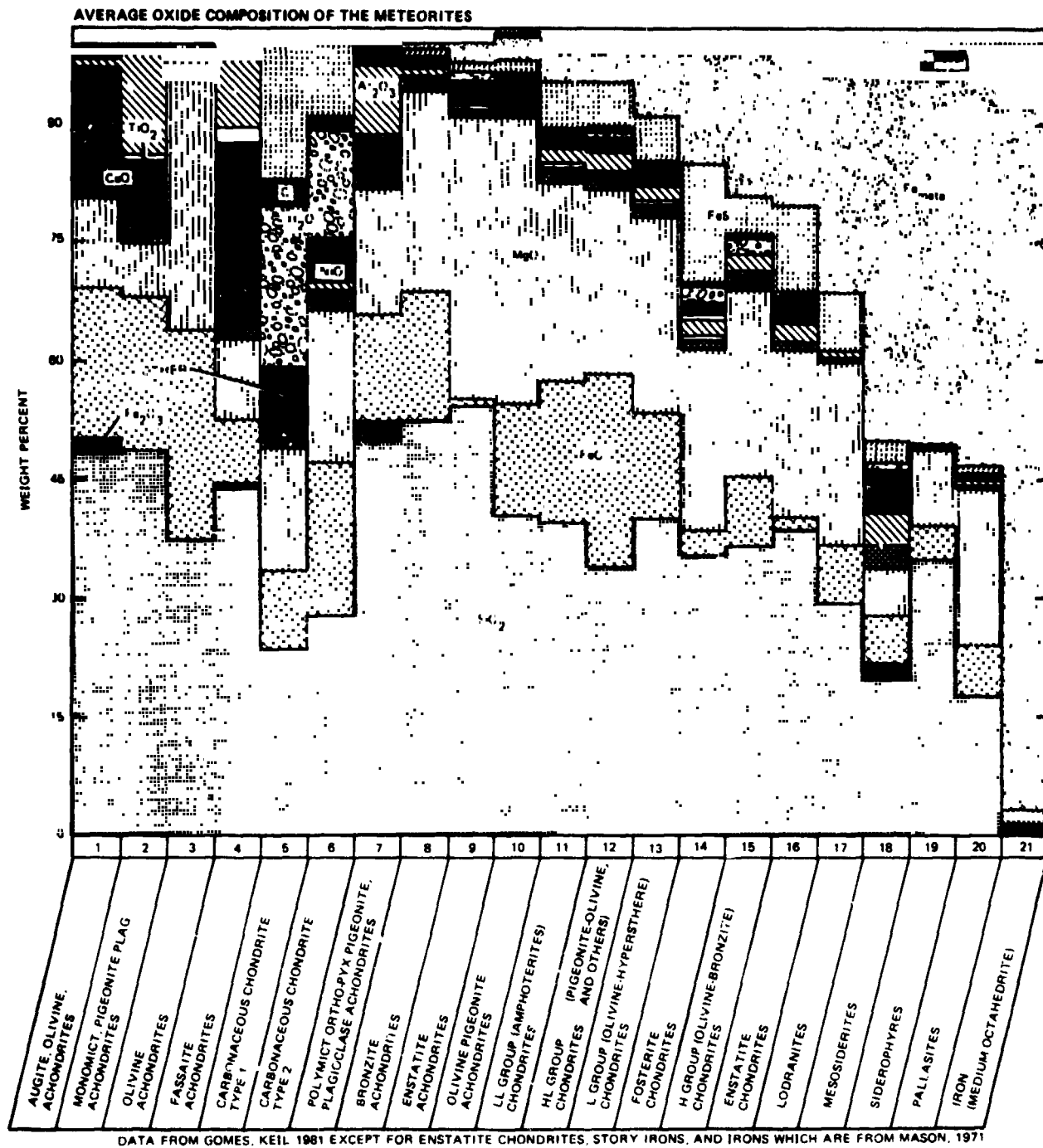
Figure III-3 Range of Oxide and Metal Composition of Meteorites
as an Indicator of Possible Asteroid Mineral Resources
(arranged in the order of increasing free metal content)

the Viking landers are probably composed of a volatile rich clay or clays, iron oxide, and fragments of weathered basalt. There is H_2O and CO_2 permafrost present. There is also an atmosphere (~5 mb) which is composed of N_2, O_2, CO_2, H_2O, CO, O_3 and other gasses.

Processing of Martian materials will be significantly different than that for lunar or asteroidal materials in several key areas. The atmosphere (~5 mb) and volatiles may make terrestrial-like processes more attractive than they are for the Moon. The low solar intensity (~½ that at Earth orbit), further attenuated by atmosphere and atmospheric dust, makes solar power a less attractive source of energy for processing.

C. MINERALS OF SPECIAL INTEREST

Each location described above has some minerals of special interest for the production of certain products. The list of minerals considered valuable will enlarge as the technology needed to utilize other minerals develops. The minerals considered as valuable materials for processing in space are as follows:

1. Volatiles for Propellants and Life Support

Hydrogen from the solar wind is present in the lunar regolith in association with fine soils rich in ilmenite onto which it is adsorbed. It can be released by moderate heating. The most optimistic estimate of the availability of hydrogen is 10 ppm (Ref. 43). The quantity available is inadequate for propellant requirements. The concentration probably will not warrant processing lunar material for the H_2 only but the H_2 will most likely be extracted if the soil is processed for any other use.

Water is very probably present in large amounts on Mars and in the Cl carbonaceous chondrites. It is also probably available in some (or many) Earth crossing asteroids. (See Appendix B.) Its availability in the form of permafrost in the permanently shaded portions of the Moon is in dispute and not to be considered a probable source due to the high heat flow on the Moon and the extreme dryness of the lunar regolith. A lunar polar satellite would be required to confirm its presence.

The availability of water is of fundamental importance in the utilization of extraterrestrial resources. The use of the readily extractable (electrolysis ~3000 kwh/ton) O_2 and H_2 for the refueling of transport vehicles will not only greatly reduce the cost of logistics, but may be decisive as to the economic feasibility of the utilization of extraterrestrial materials in general. Oxygen and water are further needed for life support in manned operations. Hydrogen, finally, may be used as a reducing agent for the extraction of metals from silicates, which widens the spectrum of feasible processes.

Carbon dioxide is plentiful on Mars and can be derived from C1 carbonaceous chondrites. Carbon is found in the carbonaceous chondrites as organic complexes and graphite, approximately a few percent by weight. Carbon is also found as nodules in Ni-Fe bodies intimately mixed with FeS. Carbon is as scarce as hydrogen on the lunar surface.

Very small amounts (~1%) of free sulfur have been found in C2 carbonaceous chondrites. Also very small amounts are evolved from the lunar soil upon heating to temperatures as low as $750^\circ C$. Troilite (FeS) is found in many asteroids (ordinary chondrites 5-6%, carbonaceous chondrites ~17%, Ni-Fe 0-5%, enstatite chondrites 5-10%) (Ref. 53, 62, 63). Troilite is rare on the lunar surface and in achondrites.

2. Silicates

Silicates have a wide application as structural materials. The plagioclase extracted from the indigenous silicates can be formed into glass components, such as plates, pipes or bubble structures, as well as into glass foam which may be useful as a building or shielding material. It is very plentiful in the lunar highlands (over 80% in the anorthosite) and is somewhat plentiful on the maria as well. Its density and low melting point may allow its separation from other materials in the liquid state (particularly in the case of oligoclase). Oligoclase is present as a minor phase interspersed in the ordinary chondrites. Anorthite is found in the achondrites and on the Moon. Plagioclase is likely to be found on Mars as well.

Basalt, and basaltic composition material can be found in many places in the inner system (the Moon, probably Mars and some asteroids). It can be cast, producing corrosion resistant tiles, pipes and vessels. Vacuum annealing may permit the production of high quality products. Cast basalt is presently used commercially in Czechoslavakian industry and others (Ref. 72).

Fine grained soil. (regolith) may be sintered and compacted to produce blocks and bricks for some structural applications. Fine soils can be found on Mars, the Moon and probably on some of the main belt asteroids.

3. Metals and Metal Oxides

Nickel-iron can be used as a structural material and for electrical and tooling applications. It can be found in ordinary chondritic material in the Earth-crosssers (about 18%) and probably in main belt bodies (up to 98% by weight). Small amounts of free iron (<1%) can be found in the lunar regolith. Its thermal, magnetic, and structural characteristics and density make it relatively easy to separate from associated troilite and silicates.

Ilmenite is found primarily in the lunar maria where it may reach concentrations of over 10%. When heated it becomes magnetic and may be separated. Ilmenite is a source of titanium, but it requires high energy processing to separate out the titanium. Ilmenite may also be found in some achondrites in minor amounts.

4. Evaporites

Sulfates and carbonates may probably be present in the Earth-crossing asteroids of C1 composition. The material may be disseminated or found as veins. This material may also be found extensively on the Martian surface.

5. Rare Earth Elements and Precious Metals

Rare earth elements are concentrated in the KREEP basalts found in the lunar highlands. Platinum Group Elements are concentrated in the Ni-Fe asteroids.

D. SOURCES FOR THE EXTRACTION OF METALS AND VOLATILES

The major sources of metals and volatiles are identified in <u>Table III-6</u>. Aluminum may be derived from anorthite in the lunar highlands and some asteroid types. Iron may be derived from the olivine and pyroxene in the maria basalts, the chondrites, some achondrites, and the Martian surface. Oxygen can be extracted from essentially any type of silicates. Hydrogen could be derived from any water-bearing body; it may also be extracted in small quantities from the lunar topsoil, where it is adsorbed from the solar wind.

Table III-6 <u>SOURCES OF METALS AND VOLATILES</u>

RESOURCES	METAL CONTENT		VOLATILES
	HIGHER	LOWER	
LUNAR SOIL	Ti, Fe, Al, Si	Mg, Ca	O_2, H_2
LUNAR HIGHLANDS	Al, Ca, Si	Fe, Mg	O_2
ORDINARY CHONDRITES	Fe, Mg, Ca, Si	Ni, Al	O_2
CARBONACEOUS CHONDRITES	Fe, Mg, Si	Ca (C)	O_2, H_2O, COH
Fe - Ni ASTEROIDS	Fe, Ni, Cu	PRECIOUS METALS	-
LUNAR PERMAFROST*			H_2O*

* SPECULATIVE - POSSIBLE BUT NOT VERIFIED

E.   USEFULNESS OF MINERALOGICAL INFORMATION

(1)   Degree of knowledge of the location in question.

	Composition	Petrologic Texture	Geologic Structure
Mars	good/incomplete	good to very poor	fair to poor
Moon	good/incomplete	very good to poor	good to poor
Earth-crossers	fair to poor	poor	fair to poor
Main Belt Asteroids	poor	poor	poor
Mars moons	poor	poor	fair to poor

(2)   Degree of value of the locations in question.

	Volatiles	Metals	Accessibility
Mars	excellent	unknown	poor
Moon	very poor	fair to poor	excellent
Earth crossers	excellent to poor	poor to excellent	fair/excellent
Main Belt Asteroids	good to excellent	maybe excellent	poor
Mars moons	may be good	unknown	good/excellent

F.   MOCK EXTRATERRESTRIAL MATERIALS STRATEGY

In order to become familiar with the raw materials of space and to
conduct experiments, engineers need material for evaluation. This presents
problems in terms of availability and expense which may be partially overcome
by preparation of simulated assemblages of extraterrestrial materials. These
materials are assembled from Earth minerals which are ground and mixed in the
appropriate proportions. The bulk mineralogy of extraterrestrial materials
cannot be matched by whole Earth rocks since the Earth rocks formed under
their own set of unique conditions. However, their mineralogy can be
approximated by mixtures of the most common constituents (olivine, pyroxene,
plagioclase, Ni-Fe, layer lattice silicates, and troilite), most of which are
fairly common terrestrial minerals. Fortunately, for most purposes, the
exotic minerals are minor constituents and can be neglected.

Where it is desired to simulate the texture of an extraterrestrial material one must consider the effects of oxygen fugacity, gravitational forces, pressure history and thermal history. It should be noted that lunar and meteoritic materials examined to date have been formed under conditions unique to their extraterrestrial environments. The texture of breccias and crystalline rocks require high temperature and/or pressure for formation. the chondrites usually contain small spheres of dry mineral which formed under unique, high temperature conditions and are difficult to simulate texturally in the laboratory. Because of the pulverized nature of the mock materials, regolith encountered on the Moon, Mars and asteroids may be the most conveniently modeled material.

The silicate melt studies currently being pursued in this program are not affected by considerations of texture of the mock extraterrestrial materials. Since the mock materials are heated above their liquidus temprature, textural considerations are unimportant and overall chemistry is the important parameter. However, texture of the mock materials is expected to play a role in the behavior of the compaction experiments. This influence is being considered in the design of these experiments. Also, these mock materials have not been outgassed of absorbed volatiles. The presence of some additional volatiles may affect their behavior in the experiments.

Three simulated materials have been prepared so far. They are lunar anorthosite, lunar basalt, and bronzite chondrite. Their composition is shown in Table III-7. The minerals were collected (some by hand, some through mineralogic supply companies), then ground, weighed, and mixed in the appropriate proportions. Grinding was done by passing small samples through a jaw crusher and then a ball mill. Contamination was minimized by cleaning the equipment between different minerals. Size distributions of the materials are shown in Figure III-4. The finer nature of the bronzite chondrite is due to the use of Ni-Fe powder which supplied the Ni-Fe.

These mock materials are used in all experimental investigations described in Section VI (Processes), specifically in

> Particle Compaction Experiments (Sec. VIA1, a and b)
> Liquid-Phase Silicate Experiments (Sec. VIA2, a to d)
> Electrolysis Experiments (Sec. VIB 2a)
> Vapor Phase Reduction Experiments (Sec. VIB 3e)

Table III-7   MOCK MATERIAL COMPOSITIONS

Bronzite Chondrite

mineral	formula	wt.%
olivine	$(Mg,Fe)_2SiO_4$	33
bronzite	$(Mg,Fe)SiO_3$	28
Ni-Fe	8% Ni, 92% Fe	20
oligoclase (Na rich)	$NaAlSi_3O_8-CaAl_2Si_2O_8$	10
pyrrhotite (troilite)	$Fe_{(x-1)}S$	5
apatite	$Ca_5(PO_4)_3(F,Cl,OH)$	4

Lunar Anorthosite

mineral	formula	wt.%
anorthite/bytownite	$NaAlSi_3O_8-CaAl_2Si_2O_8$	83
olivine	$(Fe,Mg)_2SiO_4$	16
augite*	$(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$	1

Lunar Basalt

mineral	formula	wt.%
augite	$(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$	50
anorthite/bytownite	$NaAlSi_3O_8-CaAl_2Si_2O_8$	27
ilmenite	$FeTiO_3$	20
olivine	$(Mg,Fe)_2SiO_4$	3

*   Pigeonite would have been preferable, but none was available at the time samples were made.
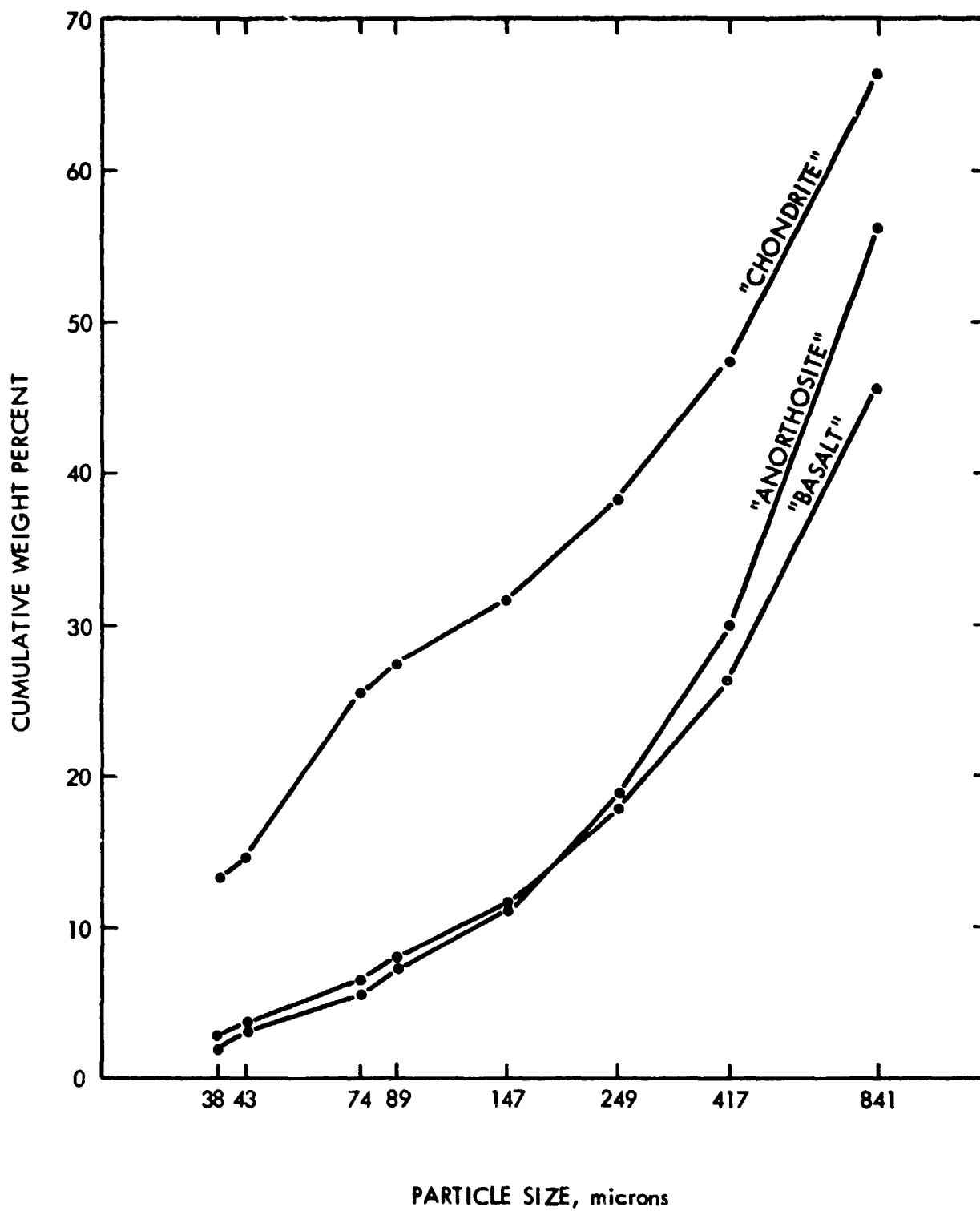
Figure III-4   Sieve Analysis of Prepared Mock Materials

Section IV

MINING AND MATERIAL CONDITIONING

Mining and Material Conditioning comprises all operations required to transfer the raw material from its native location and state into the most effective condition for subsequent processing. The relationship of mining and conditioning to the total processing sequence is illustrated in Figure IV-1. As indicated, the resulting intermediate material from mining and conditioning is designated as "feedstock" and is adapted to specific processes with regard to composition and form.

MATERIAL CONDITION OR STATE	IN-SITU MINERALS		FEEDSTOCK		PRIMARY PRODUCT		END PRODUCT

OPERATIONS	MINING & CONDITIONING	PRIMARY PROCESSING	SECONDARY PROCESSING
	• ACQUISITION • COMMINUTION • SIZING • BENEFICIATION	• EXTRACTION • SHAPING • POST-TREATMENT	• FABRICATION • ASSEMBLY • INSTALLATION
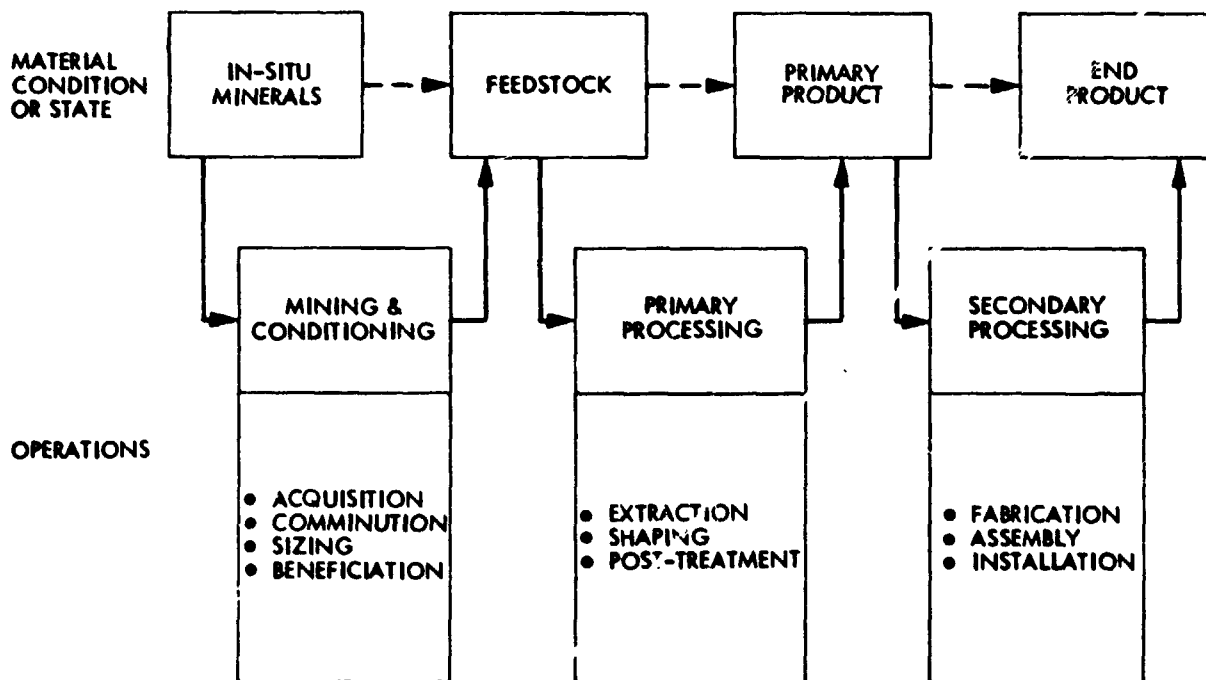
Figure IV-1 Major Processing Phases and Product Levels

A. OVERVIEW OF MINING AND CONDITIONING OPERATIONS

The initial step of Mining and Conditioning is the raw material
acquisition from the designated mining site, either in discrete quantities (in
the case of rock) or at a continuous removal rate (in the case of soil). Raw
material acquisition is the equivalent of terrestrial mining and applies to
all processes. Other steps of mining and conditioning are optional as their
applicability varies with the indigenous raw material form and with the
feedstock requirements of specific processes. They include comminution of the
acquired rock or breccia portions into fragments of a manageable size, sizing
of fragments or particles and beneficiation or enrichment of the sized
material with regard to composition.

The mining and conditioning phases, as related to the encountered raw
material form (rock, soil) are illustrated in Figure IV-2.

While all these operations except in-situ melting have well-established
counterparts in terrestrial mining, the expected extraterrestrial conditions
are likely to dictate detachment from terrestrial practices and the adoption
of space-adapted approaches.

B. ENVIRONMENTAL CONSIDERATIONS

The most significant environment in mining and conditioning is the
gravity level. The relatively high g-levels encountered on the moon and the
larger main-belt asteroids greatly simplify the movement of material in
acquisition operations, as well as movement and segregation in sizing and
beneficiation. The low g-levels of the earth-approaching asteroids in the
order of 10^{-4} to 10^{-6}g call for the development of adapted techniques,
based on induced g-forces or electrostatic methods.

Many terrestrial beneficiation, transport and process operations depend
on ready availability of working fluids (e.g., water, air). These are
unavailable or available in limited quantities (e.g., on Mars) and import from
earth is likely to be impractical except for special purposes like life
support.
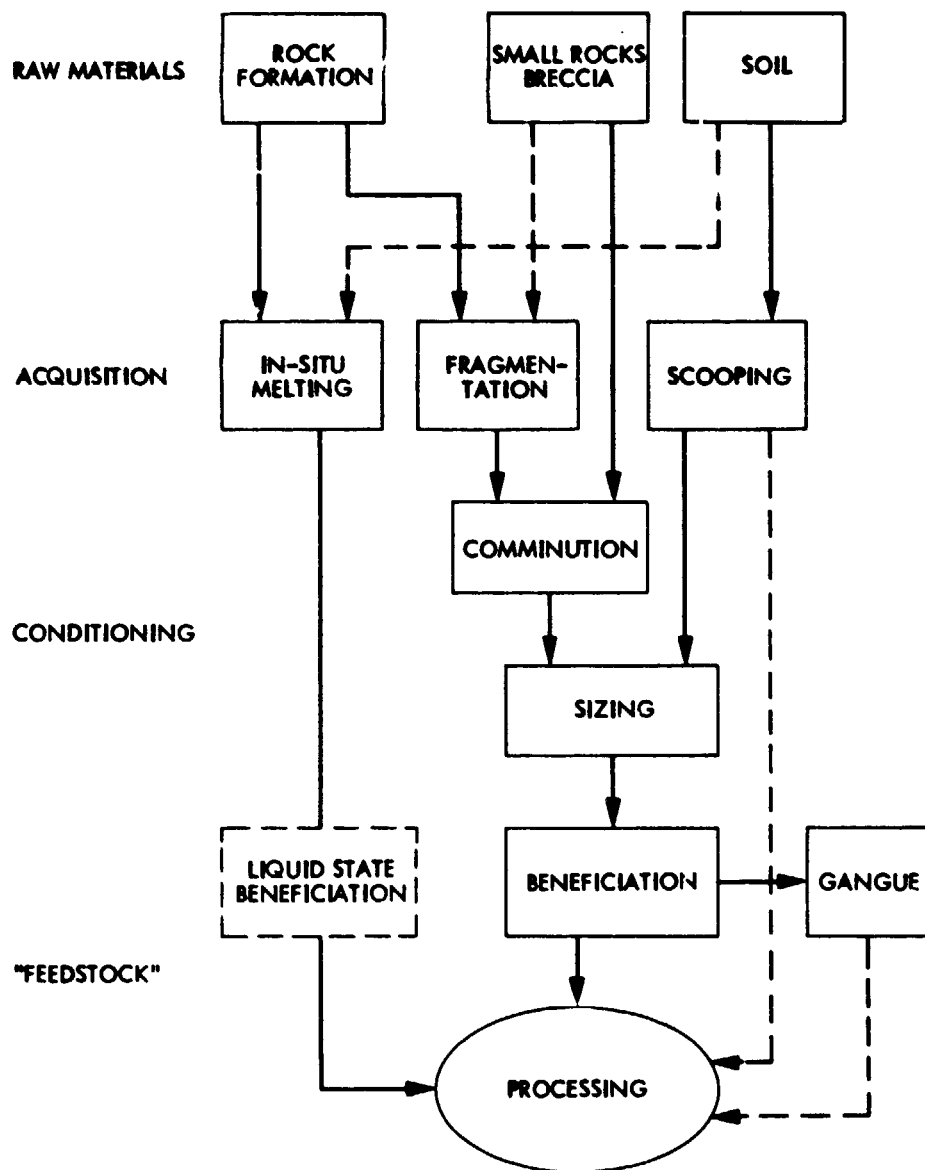
Figure IV-2 Flow of Mining and Material Conditioning Operations

The high vacuum environment, encountered in all cases, is less
significant, yet presents some problems due to the absence of adhering gases
which may enhance seizing of particles and increase tool wear. The absence of
an atmosphere will have a major impact on heat transfer associated with
processing. Vacuum further leads to mass loss by vaporization in most melting
operations.

C. SELECTION OF THE MINING SITE

It is assumed that the primary site selection, particularly the choice of
the body and the general mining region on that body has been provided by
exploration missions on the basis of such criteria as mineral resources,
accessibility, topography and the distances from the sun (solar energy flux)
and earth (transportation requirements, communications time).

The specific site selection is considered a part of the mining operations
and employs instrumentation of the mining facility upon deployment, such as
sensors for various functions. Criteria for the selection of the specific
site include the composition, form and location of resources, the size of the
useful mining area and its adaptability to other facilities, the condition of
the surface (ideally near-plane), the solar exposure of the site as related to
body rotation, and the accessibility by communications and teleoperations,
either from a spacecraft or from earth. The specific site selection may be
preceded by a detailed survey of the object from the spacecraft prior to plant
deployment.

D. RAW MATERIAL ACQUISITION

Acquisition represents the extraterrestrial equivalent of terrestrial
mining. However, the peculiar extraterrestrial conditions may call for a
distinct departure from terrestrial practices and facilities (Ref. 64, 65).
Particularly, it has to be attempted to minimize the need for complex and
massive machinery, the need for material transport and storage, the use of
tooling subjected to wear and replacement, as well as the need for human
attendance.

Terrestrial mining consists of a long sequence of individual operations.
It involves drilling, blasting, collection of rock fragments, loading,

transport and intermediate storage, each operation carried out with its own set of equipment and operators. Under extraterrestrial conditions this should be replaced by a continuous flow of operations or, ideally, by one single operational cycle, feeding the acquired material directly into the raw material conditioning system or into the processing plant.

1. Acquisition by In-Situ Melting

A typical example of a one-cycle technique is acquisition by in-situ melting, which is adaptable to all raw materials of low thermal conductivity, such as all types of silicate rocks or soil. It employs direct solar energy by means of a solar "furnace" (Figure IV-3), consisting of a heliostatically controlled paraboloid concentrator and an offset reflecting mirror (analogous to an offset-feed antenna). The molten material may be cast into ingots for further disposition or, preferably, fed directly into the processing cycle. This applies primarily to processes involving molten silicates, such as the casting or liquid extrusion of silicate components, and to processes of metal and oxygen extraction from the melt, as well as to the acquisition of water from permafrost. Besides its simplicity, in-situ melting offers the advantage of low electric energy consumption, required only for furnace attitude control and mechanical material handling systems. Problems of in-situ melting are (1) mass loss and change in composition due to gas evolution in the high-vacuum environment, (2) achievement of an appreciable melt depth as the surrounding solid material remains at low conductivity, (3) dependence on insolation intensity (solar distance) and periods (day/night) and (4) optical properties of the raw material.

It may be possible and advantageous to utilize in-situ melt beneficiation (analogous to zone-refining) to achieve desirable concentrations of specific materials.

2. Acquisition of Soil

The acquisition of soil is best accomplished by some form of scooping, preferably continuous, which has to be adapted to the existing g-level. At appreciable g-levels, as on the lunar maria, the soil may be lifted into the preferably mobile processing plant by buckets attached to an

OFFSET-FOCUS
PARABOLOID

OFFSET
MIRROR
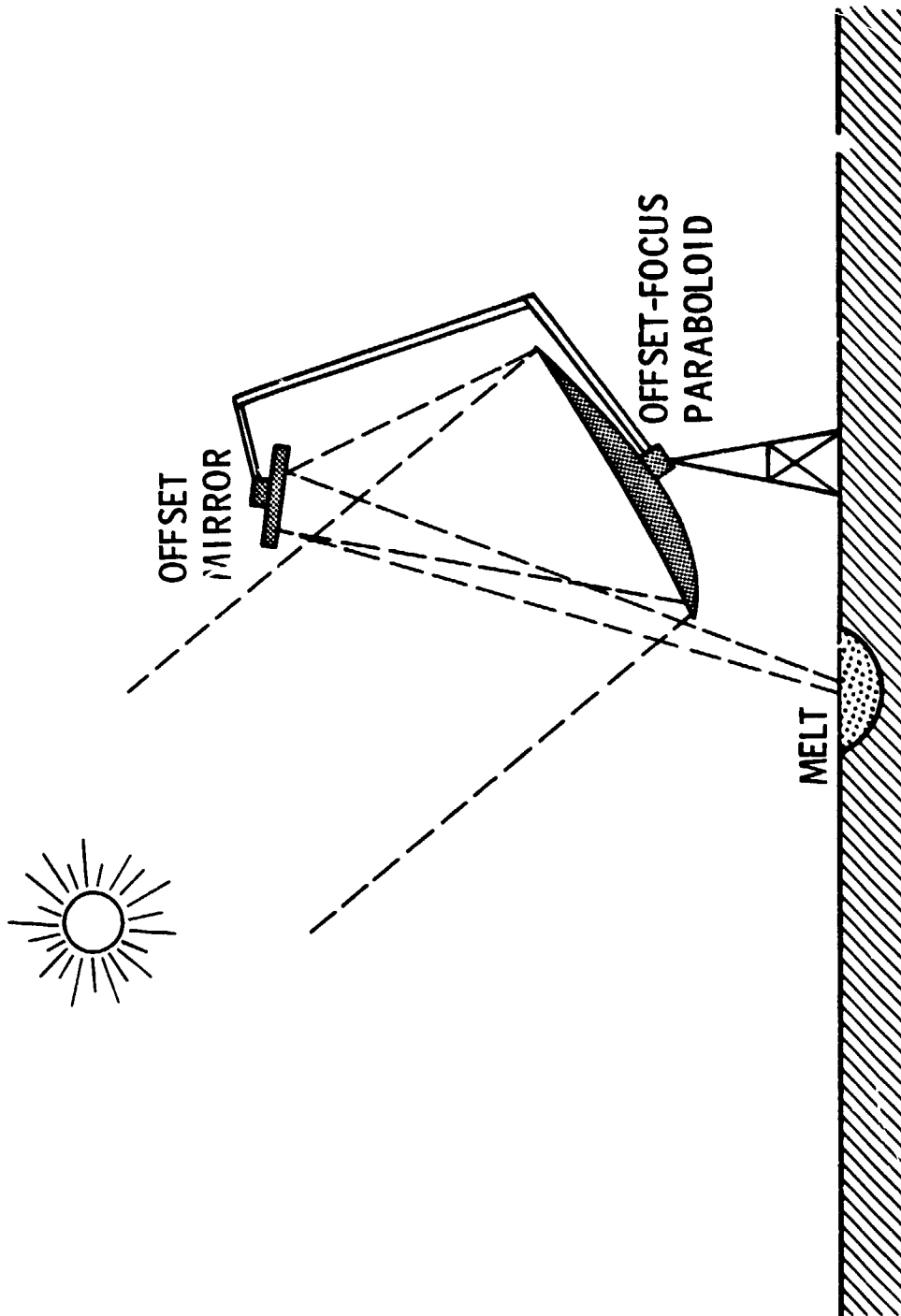
MELT

Figure IV-3 Use of Solar Furnace for In-Situ Melting

endless belt, or by a rotating helical feeder as illustrated in __Figure IV-4__.
The depth of penetration is controlled by a surface sensor system, and the
feeding rate by the processing throughput. For low g-levels the buckets of
the belt feeder are replaced by two clam-shells which open (by sensor control)
when approaching the surface and close shortly before emerging from the soil.

It is also conceivable to employ an electrostatic belt, with an
electrode of opposite polarity inserted in the soil. Electrostatic techniques
appear to be generally promising for all types of materials handling under
essentially gravitationless conditions as encountered on smaller asteroids.
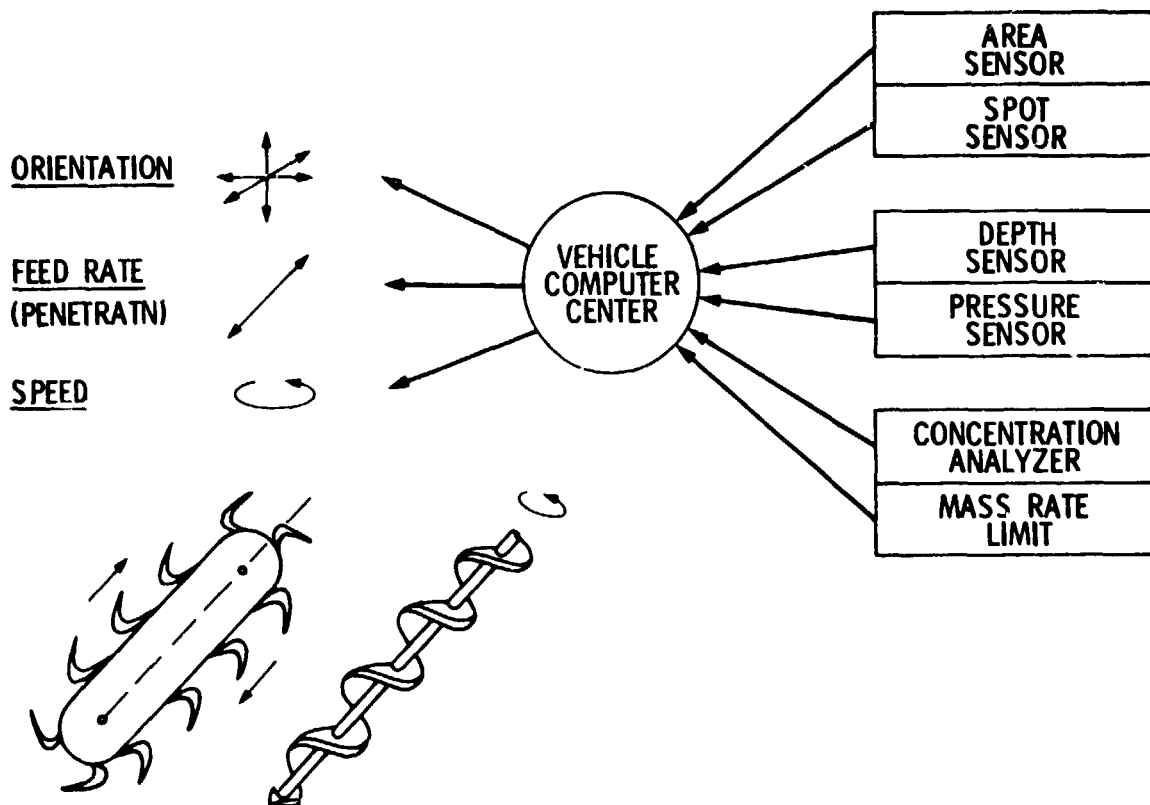
Figure IV-4 Soil Acquisition Devices and Controls

3. Acquisition from Large Rock Formations

If in-situ melting is not feasible or desirable for some reason,
more conventional methods of drilling and blasting may be acceptable with the
simultaneous use of a blast shield which prevents the fragments to be
propelled into extended trajectories. While adaptable to the moon, other

techniques have to be devised for low-g conditions. One generally applicable
method appears to be the carving-out of rock fragments by means of a laser
beam, or even a concentrated solar light beam. The laser method has the
advantage that the wavelength can be adapted to the high-absorption region of
the rock material. For silicate rocks the common CO_2 laser would be very
efficient. One problem at low g-levels is the removal of the molten material
from the cutting zone and the change of the optical properties of the raw
material upon melting. This may be resolved by sufficient beam intensity to
generate instant vaporization.
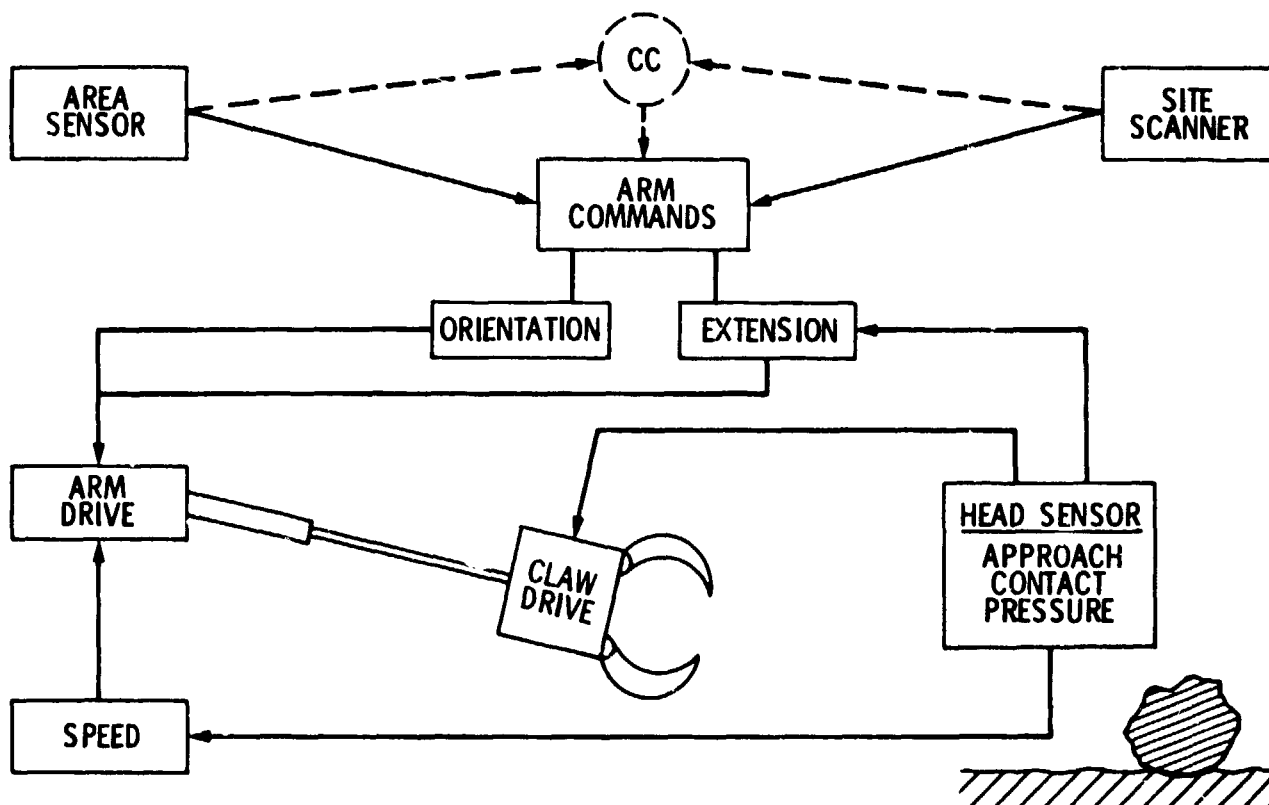
Figure IV-5 Gripping Arm for Rock Collection and Obstacle Removal

4. Acquisition of Small Rocks and Breccia

The collection of breccia or small rocks on the surface may be
accomplished with a rake-like area scooper, or with a highly maneuverable
gripping arm, controlled by sensors for object identification, approach
motion, contact and contact pressure. One conceptual design of the gripping
arm and its controls is shown in Figure IV-5. It may also be used as obstacle

remover for a mobile mining/conditioning unit. Gripping arms have the
advantage that they are not dependent upon g-level. (All scooping devices
work only at appreciable g-levels.)

5. Summary of Acquisition Techniques

The applicability of the discussed acquisition techniques to various
raw material types/forms, raw material locations and g-levels is summarized in
Table IV-1.

Table IV-1   SUMMARY OF RAW MATERIAL ACQUISITION TECHNIQUES

TECHNIQUE/DEVICE	RAW MATERIAL TYPE	MOON	ASTER.
IN-SITU MELTING	SILICATE ROCK AND SOIL	X	X
BELT/HELICAL SCOOPER	SOIL AT APPRECIABLE G	X	
CLAM-SHELL SCOOPER	SOIL AT MICRO/ZERO-G	*)	X
ELECTROSTATIC BELT	SOIL AT MICRO/ZERO-G	*)	X
RAKE (AREA) SCOOPER	BRECCIA AND SMALL ROCKS	X	
DRILLING, BLASTING	SILICATE AND ROCK FORMA-TIONS	X	
LASER CUTTING	SILICATE AND Fe/Ni ROCK	*)	X

*) APPLICABLE, BUT NOT PRACTICAL (OTHER METHODS MORE EFFECTIVE)

In-situ melting and the continuous scooping of soil emerge as
primary options. They promise a high degree of simplicity and practicality
and a wide range of applications, covering the majority of the raw material
types and forms of early interest (Moon, near-Earth asteroids). They further
exhibit low electric energy requirements and high adaptability to automation.

F.   RAW MATERIAL COMMINUTION

Practically all processes using solid raw materials require feedstock in
particle form. Where soil is not available, rock or breccias have to be
reduced to small particle sizes. Some conventional comminution methods are

illustrated - in principle - in Figure IV-6 (Reference 66). While all methods
are adaptable to space operations at an appreciable g-level, some have to be
modified for microgravity conditions. For the friable rocks typical of
carbonaceous chondrites, comminution may be accomplished in a continuous
operation using roll crushers or suspended-spindle gyratory crushers.
Comminution of hard rocks typical of the moon and the ordinary chondrites is
somewhat more difficult if extensive tooling wear is to be avoided. In this
case, jaw crushers may be more effective, as their performance is less
affected by tool wear. However, the lower limitation of fragment size
requires additional comminution to particle size by a ball mill or an
equivalent device, yet to be defined, for use in low-g environments. Another
method of comminution is impact fragmentation, by propelling the rocks at high
speed against a target, using a small mass driver for acceleration.

Comminution generally requires massive facilities and appreciable
electric power. It should, therefore, be avoided wherever soil is available
and product needs can be adapted to the specific materials which can be
extracted from this soil.

(a)   (b)   (c)   (d)   (e)   (f)
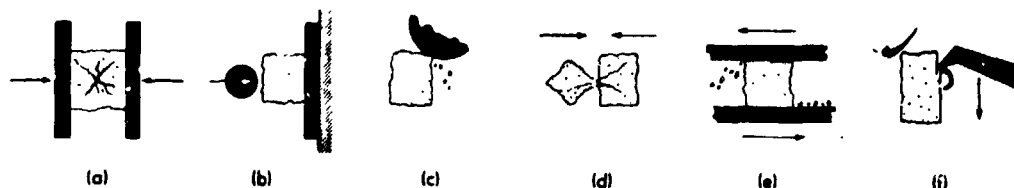
Figure IV-6 Comminution Techniques (from Ref. 66)
             (a) Compression; (b) Compression Impact;
             (c) Nibbling; (d) Self-Impact; (e) Abrasion.
             (f) Shredding

F.   PARTICLE SIZING

The feedstock requirements of most processes as well as product quality
considerations call for particles of a discrete size range, a specific size
distribution or a maximum size limit. The sizing or volumetric sorting of
particles applies to soil as well as to the end product of the comminution
process. Sizing is a necessary feedstock conditioning operation for: (1)
silicate compaction and powder metallurgy processes to assure high product
quality (strength, fracture resistance); (2) vapor-phase reduction processes
to assure complete feedstock vaporization; (3) chemical processes to enhance

chemical reactions and to reduce the consumption of imported fluids. In a gravity environment (moon) sizing can be accomplished by conventional methods using a set of oscillating screens, better known as sieving. For an essentially gravitationless environment, appropriate methods have yet to be devised. Electrostatic techniques, again, appear most promising.

G.   RAW MATERIAL BENEFICIATION

The objective of beneficiation is to generate a feedstock with the highest possible concentration of the minerals essential for a specific process and/or product. It is practically mandatory for all processes of metal and oxygen extraction. It applies also to certain silicate products and related processes, particularly to the production of glasses or other nonmetallic products with specific property requirements.

Like the previously discussed operations of mining and conditioning, beneficiation is preferably carried out in the solid ("dry") state, since the use of fluids as practiced extensively in terrestrial beneficiation is highly undesirable for the following reasons: (1) practically all applicable fluids have to be imported from earth; (2) most fluids are subject to serious or catastrophic loss in vacuum in the event of a leak or micrometeorite puncture of equipment; (3) cleaning and/or recycling of fluids is expensive in terms of facilities and operations. So far, two methods of dry beneficiation have been conceptually defined: magnetic and electrostatic separation. The possibility of other techniques based on size, shape or other physical characteristics which may be related to composition will be examined. (For example, the concentration of alkali metal oxides is higher in the lunar fines than in the bulk soil).

1.   Magnetic Separation

The simplest method of magnetic separation is the recovery of magnetite which can be accomplished by feeding the raw particles through the field of one or several permanent magnets. Figure IV-7 shows one type of magnetic separator designed to separate the magnetic minerals from the non-magnetic minerals. For concentrating magnetite, a low intensity magnet

FEED RAW MATERIAL
HERE

FEEDING HOPPER

POLE PIECE

INDUCED ROLL

BRIDGE
BAR

COIL

Figure IV-7   Magnetic Particle Separator

1ST MAGNETIC
PRODUCT

INDUCED ROLL

POLE PIECE

2ND MAGNETIC
PRODUCT   NON-MAGNETIC
            PRODUCT
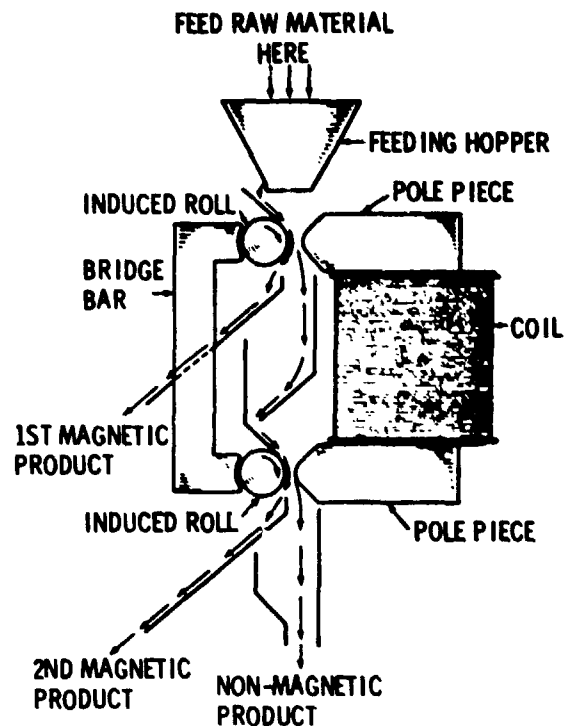
can be used because the mineral is strongly magnetic. In treating less
magnetic materials, a high intensity magnet or an electromagnet may be used.
This system can be automated for use on the moon. While feeding in an
appreciable gravity environment presents no problems, appropriate methods for
microgravity conditions (asteroids) have yet to be defined.

2.   Electrostatic Separation

          Electrostatic separation is more complex, but offers significant
advantages, such as independence of magnetic mineral properties and
adaptability to gravitationless conditions. This process is utilized to
separate conducting, insulating and semiconducting materials. Figure IV-8 is
a schematic diagram of a high-tension e~ ~ctrostatic separator (Reference 67).
A is a conducting drum of metal which turns in the indicated direction. This
metal drum is grounded. C is an electrode which is maintained at a high
potential with respect to A; C is usually negative. A thin layer of minerals
is fed onto the top of the roll and follows it around. The discharge from the
point electrode tends to polarize the mineral particles, i.e., drive their

-42-
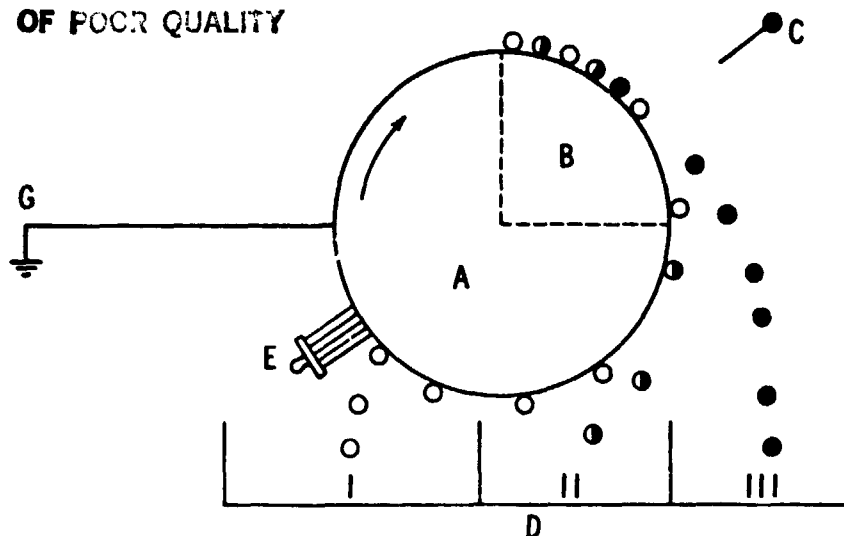
Figure IV-8 Electrostatic Particle Separator

A.   Grounded Metal Drum
B.   Separating Zone
C.   Comb Electrode (Usually Negative)
D.   Collecting Bins
E.   Brush or Scraper
F.   Ground Connection

Conducting particles collected in compartment III;
Nonconductors in I; Middlings in II.

electrons away from C and towards A.   In conducting minerals the electrons can
flow easily, and good conductors immediately lose electrons to the roll.
This leaves them with a positive charge and they are repelled by the
positively charged roll, so that they fall into the container III.   Insulators
or dielectrics retain their polarity for an appreciable length of time and
remain pinned to the roll by electrostatic action, because the side next to
the roll is negative and is attracted by the positively charged roll.   These
are carried around until they are swept off by the brush E, and dropped into
container I.   Semiconductors will follow a path so they drop into container
II.   Native metals and sulfides are conductors or semiconductors, while
silicates and some oxides are nonconductors.

This method is interesting because theoretically it represents a
"universal" concentrating method.   Almost all minerals show some difference in
conductivity.   Therefore, it should be possible to separate almost any two
minerals by this process.   High tension separators are commonly used in
conjunction with magnetic separators for resolving heavy sand mixtures into

their component minerals. The above described method can be easily automated and applied for use on the moon. However, it has to be modified for application on asteroids. More specific electrostatic separation techniques have been proposed by Williams et al. (Reference 68). Reference (8) advocates the beneficiation of materials on the moon by electromagnetic mass drivers powered by solar-electric energy.

Section V

PROCESSING — BASIC CONSIDERATIONS

A. DEFINITION OF PROCESSING

Processing is defined for this project as comprising all operations
required to convert the feedstock, as produced in the mining and conditioning
phase, into an end-product. As indicated in Figure V-1, it consists of two
major phases:

(1) Primary processing which advances the feedstock to the "primary
product".

(2) Secondary processing which comprises all operations to produce a
useful end-product from one or several different primary products.

Primary processing includes material extraction and, if applicable, product
shaping and post-treatment. The product resulting from primary processing may
be in the form of bulk material or in the form of a basic shape. Secondary
processing includes fabrication, such as final machining of the individual
products, assembly of any number of like or unlike primary products into a
subsystem and, finally, installation into the user system. The end-use status
may be attained at any point of the entire processing sequence, from the bulk
form as in the case of fuels, to a fully installed system assembly.

The schematic representation in Figure V-1 does not necessarily imply a
storage period or transport from one site to another between major operations
as is frequently the case for terrestrial operations. While such storage or
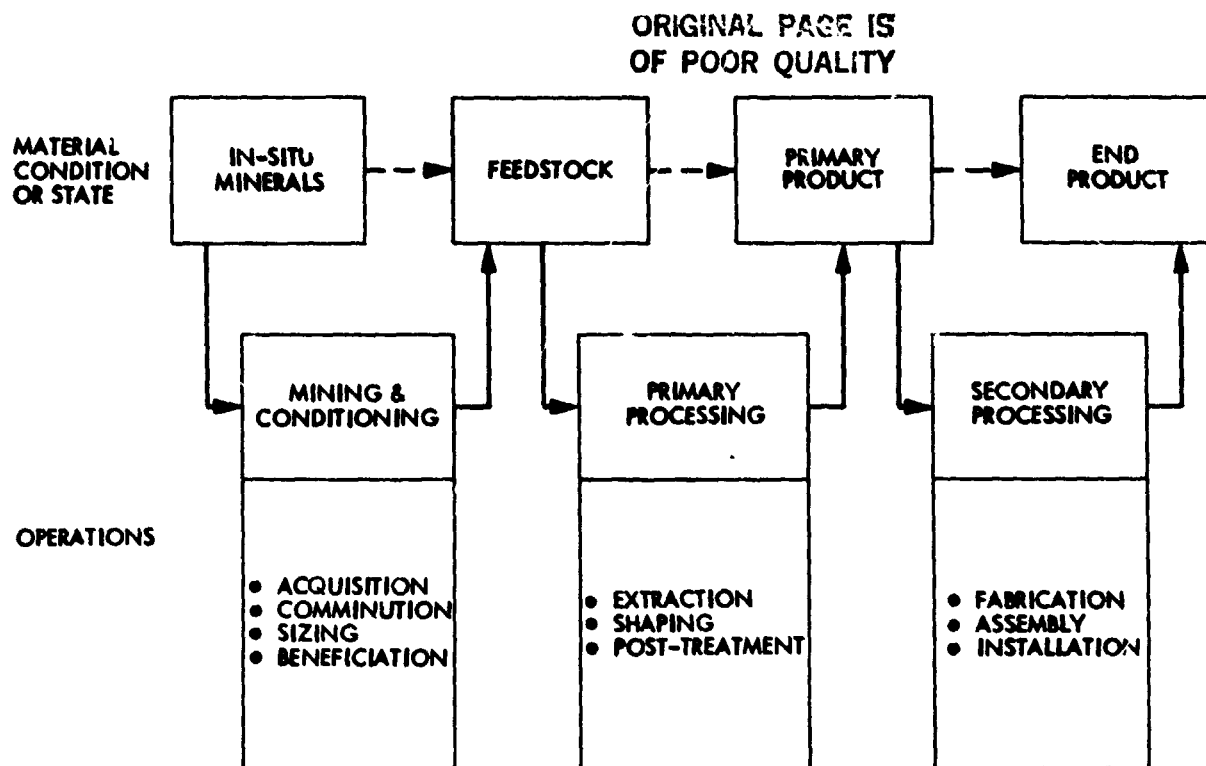
Figure V-1 Major Processing Phases and Product Levels
(Identical to Figure IV-1, repeated here for convenience.)

transport may occur, simple end-to-end from in-situ minerals to end-product
process sequences are most likely, at least for the initial phases (first few
decades) of extraterrestrial materials processing.

B. SPACE ENVIRONMENT AND ITS EFFECT ON PROCESSING TECHNOLOGY

The operational environment of space is quite different from the
terrestrial environment. It is characterized by: (1) The complete absence of
fluids, particularly an atmosphere. This condition, referred to below as
vacuum, applies at least to the moon, the asteroids and high-earth-orbits
where the pressure is in the order of 10^{-14} torr. (2) Low gravity, ranging
from 0.2 g on the moon to 10^{-5} g on smaller asteroids and essentially zero-g
in high earth orbits. (3) Solar energy unrestrained by atmospheric absorption
(except on Mars), with a flux intensity ranging from 1.35 kW/m^2 in the
near-earth region to 0.2 kW/m^2 in the main asteroid belt. (4) Variable
surface temperatures, as related to location on the body and to body
rotation. Equatorial surface temperatures on the moon, for example, exhibit a
ΔT of 290°C ranging from $+150^{\circ}$C at lunar high noon, to -140°C at
midnight.

The environment in terms of material resources, such as the scarcity of carbon and hydrogen, is omitted here since it has been included in the discussion of raw materials; it is, however, an important consideration in processing technology.

These peculiar environmental conditions call for a distinct detachment from conventional processing concepts which are extensively based on the terrestrial environment, particularly on the availability of carbon, coal and oil, the abundance of free fluids, such as air and water, as well as a high g-level. It calls for a high degree of adaptation of processing techniques and related operations to the set of environments encountered at a specific location. Moreover, it should be attempted to use the space environment to advantage, particulary the unlimited high vacuum which is extremely difficult to produce and to maintain on earth. The following discussion outlines some of the major effects of individual environments on, and the potential utilization in, processing concepts and operations.

(1) Vacuum. The reduced boiling temperatures of materials in high vacuum together with the unlimited "pumping capacity" of space permits new approaches to extractive metallurgy and even the use of sustained plasmas. Vacuum is further beneficial in powder metallurgy and the compaction of oxide particles due to the enhanced interface bond in the absence of absorbed gases. Negative effects include the mass loss in melting operations due to accelerated gas evolution and the increased friction and wear at moving surfaces. Vacuum further renders all processes employing pressurized systems impractical, as a leak would result in the immediate loss of all volatile fluids. This is particularly critical if the working fluids have to be supplied from earth.

(2) The absence of free fluids, such as water or air, precludes all conventional methods of material transport or separation using a fluid medium. The handling of solid material, such as the transfer or beneficiation of particles, has to be carried out exclusively in the "dry" state.

(3) Low Gravity. The appreciable gravity prevailing on the moon and large (main belt) asteroids is beneficial in material handling operations and in such processes as casting or segregation, permitting the use of conventional techniques with only minor modifications. Microgravity conditions, as encountered on all earth-approaching asteroids, call for entirely new approaches to these operations, yet to be determined. On the other hand, microgravity can be used to advantage in processing and may lead to new techniques, unfeasible on earth, such as liquid extrusion or contact-free drawing of shapes from a melt. In this case, typical microgravity locations, such as earth or lunar orbit, may be selected for processing operations or specific processing phases. Many microgravity processes, presently under development in various Materials Processing in Space (MPS) programs, may be directly applicable to processing of extraterrestrial materials.

(4) Solar Energy may be used directly for heating by means of a solar furnace, or converted to electric energy by an appropriate solar power system. Wherever possible, processes should be adapted to the use of direct solar energy. The sole shortcoming of the use of solar energy is the operational dependence on the day/night periods of rotating bodies and, for earth-approaching asteroids, on orbital characteristics (variable intensity). For solar-electric power the day/night restriction may be eliminated if a point of permanent insolation can be found for plant location, such as the polar regions of the moon.

(5) Surface Temperatures. Of prime interest are the low temperatures encountered during occultation periods or at the bottom of deep valleys. Besides the application in cooling systems for processing and power generation facilities, the embrittlement of materials at low temperatures may enhance such operations as raw material comminution (Section IV-E) or the removal of a silicate crust from free metal particles. It is conceivable that such processes are carried out in night operations only.

It is concluded that the beneficial effects and the applications potential of the operating environments outweigh their disadvantages. The most powerful and unique environmental resources of space are undoubtedly the unlimited high vacuum and microgravity, both heretofore not available to man. Their application in space processing promises to impart a new dimension to materials technology in general.

C.   SPACE ADAPTATION OF PROCESSES

The first and most decisive step toward extraterrestrial processing is a complete detachment from terrestrial approaches. As the envisioned operations are removed from earth, so has to be our thinking. The term "adaptation" refers to the selective use of the scientific knowledge and of the technological arts which we have accrued on earth, rather than to the attempt to modify terrestrial concepts for transfer into the extraterrestrial domain which could lead to failure. Some specific approaches to space-adapted processing are as follows:

(a) Minimize or eliminate the need for imports, i.e., the supply of consumables from earth, such as chemicals serving as reagents or water. Deliveries from earth should be confined to the processing facility power plant and the related support facilities.

(b) Utilize to the fullest extent unique extraterrestrial environments, such as the inexhaustible ultra-high vacuum, solar energy and low gravity.

(c) It should be attempted to achieve processing in one single cycle, from raw material acquisition to the primary product. This eliminates intermediate storage and transfer operations, as practiced on earth, which increases equipment complexity and energy requirements.

(d) With the exception of the compaction of regolith into silicate products, most processes involve the liquid and/or gaseous state. This favors raw material acquisition by in-situ melting

and product shaping by either casting, drawing, extrusion or vapor deposition. In compliance with (c) this permits the performance of all processing steps in one single thermal cycle.

(e) Extraterrestrial processing should employ a high degree of automation, machine intelligence and robotics. The need for automation may be considered an extension of postulate (a), above (minimization of imports), since man and his support requirements represent an extremely expensive import. Even for the rather accessible moon, a strong case for total automation, including equipment self-replication, was presented by the Santa Clara Summer Workshop on Automation (Ref. 40). The arguments for automation are even more valid for more distant objects, such as the asteroids. This automation further facilitates a gradual phasing of unmanned exploration mis· into operational missions in an unmanned mode.

(f) Processing and all related operations have to be adapted to long unattended periods between production start and the pick-up of products. In lunar operations, this time is determined by the frequency of transport traffic. For asteroids, this interval is quite long and dictated by the orbital characteristics. For low total Δv missions, plant deployment will preferably occur near perihelion. While production may be continuous, the stay time of the transport vehicle is between 5 and 10 months.

D. PROCESSING PHASES

An overview of the major operational steps involved in the processing from raw material to primary product is presented in Figure V-2. It applies to all processes based on silicates as raw material and such products as silicate components, metal shapes, oxygen, hydrogen and other gases. Silicate products may be obtained from rock by in-situ melting and liquid-state forming (at left) or from soil by either liquid or solid state (compaction) processing
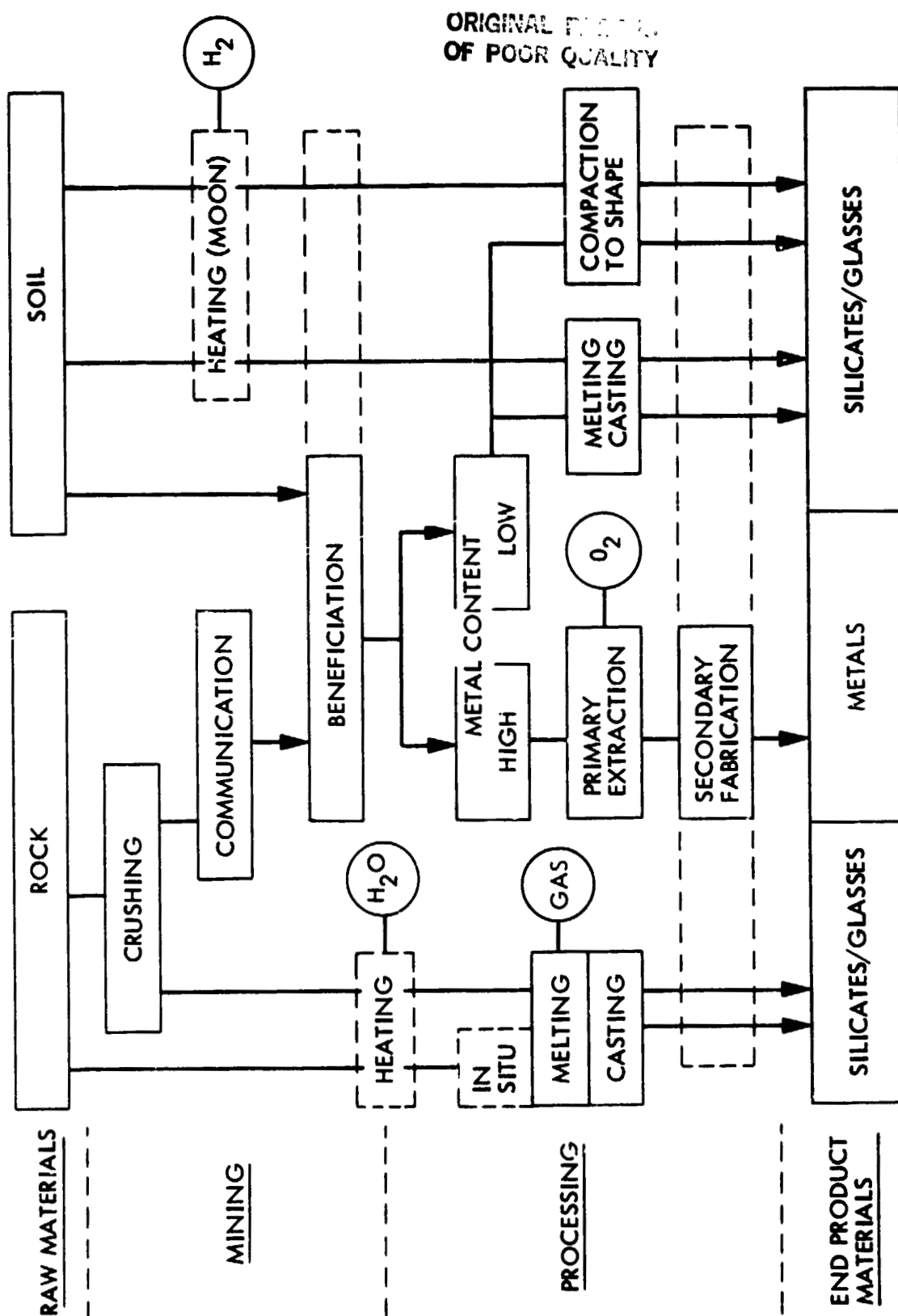
Figure V-2   Flow of Processing Operations from Raw Material to Product

(at right). The feedstock for metal products (center) is obtained by the mandatory beneficiation of soil or comminuted rock. The metal-rich portion represents the feedstock for metal extraction and shaping processes, while the remainder is either treated as waste, or as particle feedstock for silicate products (at right).

The operational steps for an example group of individual process sequences are identified in more detail in Table V-1. The dark circles represent mandatory steps and the light circles optional steps. The operation which results in the primary product is identified by a dark square. The individual processes referred to in this table are discussed in Section VI.

E.   CATEGORIZATION OF PROCESSES

The design and development of processes calls for utmost adaptation to four basic conditions and requirements:

(1) Available raw materials
(2) Desired or attainable products
(3) Environmental conditions
(4) Operational constraints

In a first-order categorization, primary processes are identified by the utilized raw materials as input and the attainable products as output (1 and 2 above). Adopting this generic identification, processes are divided into the following categories:

(A) Silicate products from silicates
(B) Metals and oxygen from silicates
(C) Metal products from free metals
(D) Hydrogen from lunar soil
(E) Fuels from bound or free water and hydrogen
(F) Carbon from carbonaceous chondrites

Present efforts are concentrated on categories A and B. Preliminary studies on C and D are in progress. Categories E and F are deferred for the

Table V-1 REQUIRED OPERATIONAL STEPS FOR VARIOUS PROCESSES

PROCESSES	RAW M. ACQUISITION	COMMINUTION	BENEFICIATION	PROCESSING	SECONDARY FAB.	POST TREATMENT
COMPACTION - COLD PRESS + HT	●			■		○
COMPACTION - HOT PRESS	●	○	○	■		
COMPACTION - IMPACT	●	○	○	■		
SILICATE CASTING	○			■		○
SILICATE LIQUID EXTRUSION	○			■		○
DRAWING - RAW M. AS IS	○		●	■		○
DRAWING - OLIGOCLASE	○	○		■		○
ELECTROLYS. - O_2 ONLY	●	○		●	■	
ELECTROLYS. - METALS + O_2	●	○	○	●	■	
PLASMA I METALS + O_2	●	○	●	■	○	
PLASMA II METALS + O_2	●	○	●	●	■	
CHEMICAL - CARBOCHLOR.	●	○	●	●	■	
CHEMICAL - OTHERS	●	○	●	●	■	

● MANDATORY STEPS   ○ OPTIONAL STEPS   ■ SHAPED PRODUCTS

following reasons: (1) The presence of free water in the form of permafrost in the polar regions of the moon is highly questionable; (2) the extraction of bound water and carbon is limited to the asteroids and requires extensive facilities.

Section VI

PROCESSES

This section gives an account of the work performed to date on individual processes. It comprises the evaluation of previously proposed concepts, the development of new concepts, the related theoretical and experimental investigations as well as the definition of the envisioned products. Process effectiveness is addressed only in general terms since a more accurate assessment would have to include quantitative data on all support operations, such as transportation and power generation which are outside of the scope of this study.

This is an annual report; the cutoff for technical contents was calendar date, not technical status. As a result, the experimental work reported in this section is the initial phase of a longer term overall planned set of investigations.

The numerical identification of processes follows the classification established in the foregoing section. The processes discussed in this section are as follows:

A. Processing of Silicates and Silicate Products.
   A-1 Particle Compaction Processes
   A-2 Liquid Phase Processes

B. Extraction of Metals and Oxygen from Silicates.
   B-1 Chemical Processes
   B-2 Electrolysis of Silicates
   B-3 Vapor Phase Reduction Processes

A. PROCESSING OF SILICATES INTO SILICATE PRODUCTS

The processes which can be utilized to produce products directly from the silicates are simple, straightforward and require only a few steps between mining and the finished product. Also, no consumables are required from Earth*. Components produced directly from the silicates can be used in fabrication of extraterrestrial structures. These structures include habitats, factories, equipment and orbital stations. Specific products which have been proposed by various investigators (Ref. 30, 69-71) include:

Structural Members	Tiles, Slabs, Blocks
Building Materials	Pipes
Transparent Glass Sheet	Containers
Foamed Glass	Basalt and Glass Fibers
Radiation Shielding	Woven Fibers
Solar Cell Backing	Electrical Insulators
Solar Cell Covers	Road Surfacing

Methods for forming silicates selected for evaluation in this phase of the investigation are (1) particle compaction, and (2) production directly from the liquid phase. The particle compaction techniques are (a) sequential application of pressure and heat, (b) simultaneous application of pressure and heat, and (c) impact forming. The liquid phase production methods are (a) pouring molten material into a mold, (b) forcing molten material through a die or orifice, and (c) pulling molten material through a die.

The extraterrestrial particle compaction techniques can be thought of as similar to the terrestrial forming processes (a) pressing and sintering, (b) hot pressing, and (c) impact compaction, respectively. Likewise the extraterrestrial liquid phase forming techniques can be thought of as similar to the terrestrial forming methods (a) casting, (b) extrusion and (c) drawing, respectively.

1. Particle Compaction Studies

The major characteristics of particle compaction techniques are

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* An exception may be the production of foamed glass where the import of foaming agents from Earth may be required.

presented in <u>Table VI-1</u>. The raw material in each case (sequential pressure + heat, simultaneous pressure + heat, and impact) is extraterrestrial silicate rock and soil. The process concepts for each of these three methods are derived from the terrestrial methods (a) dry pressing and sintering, (b) hot pressing and (c) impact forming by particle-particle bonding, respectively. The equipment requirements for the particle compaction methods are few. A means of separating various particle size fractions will likely be required. The coarse fraction of the granular material can be removed and the size range of material which is fed to the compaction machine can be adjusted to provide optimum packing density. Means of compacting the graded, particulate material (compaction machines) and means of applying heat (for processes a and b) are required in order to produce particulate compacts with sufficient strength to survive the intended service environment.

Processes a and b may utilize solar radiation for heating while impact forming will not require an external heat source. Energy to power the equipment used in the particle compaction processes may come from solar or nuclear power plants.

The products which can be formed by particle compaction methods range in size from miniature components of less than 1cm (0.4 in) in length to large blocks which may measure 3m (10 ft.) or more across. The maximum size of components formed by sequential pressure + heat is limited by heat transfer considerations, by the size and mass of compaction machines and by the ability to handle large shapes having low strength. The forming method utilizing simultaneous application of heat and pressure lends itself to the production of dense, strong components which have good resistance to wear, impact and chemical attack. Small sizes, 1cm (0.4 in) in length, can be made without difficulty. The maximum size is limited by the ability to apply the necessary pressure on the component (generally in the range 3.4 to 34 MN/m^2 or 500 to 5000 psi) and by the thermal conductivity of the granular material to be compacted. Equipment size and mass requirements affect the load available for compaction. The compact size and conductivity affect the amount of time required to heat to some minimum temperature in order to get sufficient diffusion bonding to take place. The maximum size of

Table VI-1 MAJOR CHARACTERISTICS OF SILICATE COMPACTION PROCESSES

	SEQUENTIAL PRESSURE + HEAT	SIMULTANEOUS PRESSURE + HEAT	IMPACT
RAW MATERIAL	SILICATE ROCKS AND SOIL	→	→
PROCESS CONCEPT	DRY PRESSING AND SINTERING	HOT PRESSING	PARTICLE-PARTICLE BONDING DUE TO HIGH CONTACT PRESSURE IN VACUUM
EQUIPMENT REQUIREMENTS	PARTICLE SORTER, PRESS, FURNACE	PARTICLE SORTER, HOT PRESS	• SINGLE IMPACT PRESS • MULTIPLE IMPACT PRESS/EXTRUDER
HEATING	DIRECT SOLAR	→	NONE
ENERGY SOURCE	SOLAR CONCENTRATOR, PHOTOVOLTAIC, NUCLEAR	→	PHOTOVOLTAIC, NUCLEAR
PRODUCTS	"BRICKS", SMALL COMPONENTS	SPECIALTY PARTS	(?)
ADVANTAGES	SIMPLICITY, ADAPTABLE TO AUTOMATION, CONTINUOUS, LOW ENERGY REQUIREMENTS, PRODUCE LARGE NUMBERS OF PARTS	COMPARATIVELY SIMPLE, ADAPTABLE TO AUTOMATION, LOW ENERGY REQUIREMENTS	SIMPLICITY, ADAPTABLE TO AUTOMATION
DISADVANTAGE	LIMITED SHAPE CAPABILITY	→	→

components produced by impact forming methods is primarily limited by
equipment requirements. It is not known at the present time what the
upper limit on dimension of parts may be.

The advantages of these three forming methods are generally the
same. They are inherently simple, are adaptable to automated production
facilities, and require little energy compared to most other processes.
These particle compaction methods provide a means of producing large
numbers of parts having the same dimensions in a continuous operation.
The method utilizing sequential application of pressure + heat is
particularly suited for producing components at a rapid rate. The use of
simultaneous application of heat + pressure and of impact is best
reserved for production of specialty parts requiring properties not
obtainable from the sequential pressure + heat method.

The objective of the initial particle compaction studies was to
determine the effect of process variables on the densification of mock
extraterrestrial materials. The density, specific gravity, and porosity
are determined as a function of temperature and pressure. The range of
process variables utilized in this portion of the investigation is shown
in Tables VI-2 and 3 for sequential pressure + heat and simultaneous
pressure + heat methods, respectively.

a.   Sequential Application of Heat arJ Pressure.

Experimental Procedure. Mock material mixtures were prepared as
simulants of extraterrestrial silicates. Three different compositions
were selected as covering a wide range of chemical compositions. They
represent two types of materials brought back from the lunar surface and
one which represents the expected composition of an asteroid. The sample
simulating the composition of lunar highlands was made up primarily of
bytownite, the lunar maria was represented by a basaltic composition made
up primarily of augite and the asteroidal material was represented by an
ordinary chondrite which contained primarily olivine and bronzite
(hypersthene). The mineralogical make-up of these mock materials is
identified in Table III-8.

Ten grams of each of the three mock extraterrestrial material
mixtures were mixed with 5 weight percent of a 10 percent solution of

Table VI-2 EXPERIMENT SCHEDULE FOR THE STUDY OF SEQUENTIAL APPLICATION OF PRESSURE AND HEAT

Time	Temperature, °C			
sec	1100°	1180°	1250°	1350°
60	A,B,C			A,C
600	A,B,C	A,B,C	A,C	A,C

NOTE: The letters A, B, C refer to use of mock anorthosite, mock basalt and mock chondrite, respectively.

Table VI-3 EXPERIMENT SCHEDULE FOR THE STUDY OF SIMULTANEOUS APPLICATION OF PRESSURE AND HEAT

Temperature °C	Time min.	Applied Pressure, MPa (psi)	
		6.89 (1000)	27.56 (4000)
1050	60		A, B, C
1110	60		A, B, C
1125	60	A, B, C	
1180	1	B	
1215	60		A, B, C
1315	60		A, C
1350	1	A	
1385	1	C	
1400	60		A, C

NOTE: The letters A, B, C refer to use of mock anorthosite, mock basalt and mock chondrite, respectively.

polyvinyl alcohol in water as a binder. Each material mixture plus binder was pressed at 69 MPa in a steel die set into a 2.5 cm diameter pellet. The specimens were dried at 80°C overnight. One specimen of each mock material was placed in the furnace, one on top of another, with sintered alumina beads between them. A refractory-metal-foil cup (either tantalum or molybdenum) was placed between the specimens and the tungsten hearth of the furnace.

The furnace* used in this study employed a tungsten heating element in a water-cooled stainless steel shell. The furnace could be operated in a vacuum, reducing or inert atmosphere. The samples were placed in the furnace and the furnace was sealed and evacuated to approximately 10^{-2} torr. The furnace was then backfilled with helium, pumped back down to 10^{-2} torr and again backfilled with helium. The furnace was then heated to approximately 500°C over a 2 hour period and then to the temperature of interest at a rate of 600°C/hr. The specimens were held at temperature for 1 hour then cooled by shutting off power to the furnace. Temperatures employed in this study ranged between 1100° and 1350°C.

Results. The analysis of the mock extraterrestrial materials formed by sequential application of pressure and heat is not yet complete. Data on weight loss and shrinkage during the sintering (heating) process are currently being evaluated. Evaluation of the microstructure and the nature of the crystalline phases of the sintered specimens is being performed via optical microscopy. X-ray diffraction will also be used for identification of the crystalline phase content of the specimens. The results will be presented at the end of the next reporting period.

b.   Simultaneous Application of Heat and Pressure.

Experimental Procedure. A graphite resistance heating element hot press** with a water-cooled stainless steel shell was used for the

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*   Astro Industries, Inc., Model 110GV 3580W1.

** Astro Industries, Model HP50-5080.

simultaneous application of heat and pressure experiments. The furnace
is capable of operation at temperatures to 2100°C in a vacuum, reducing
or inert atmosphere.
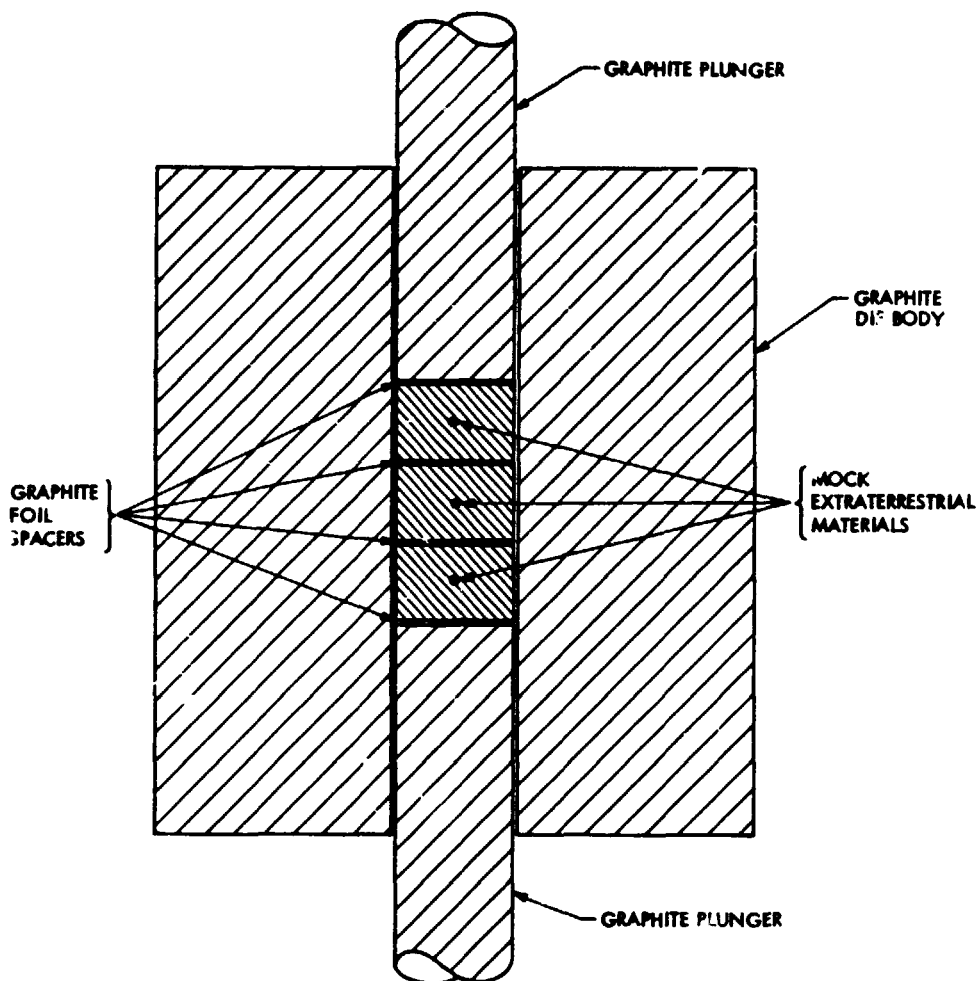
Figure VI-1   Graphite Die Assembly Used in Compaction
Experiments Under Simultaneous Application of Heat and Pressure

A graphite die and plungers were used in this investigation. Ten
gram samples of each of the mock extraterrestrial materials were loaded
into the graphite die as illustrated in Figure VI-1. The samples were
separated by graphite foil spacers. The die assembly was then placed
into the hot press and a nominal load applied. The hot press was
evacuated to a level of approximately 125 mb and then backfilled with
argon. This procedure was repeated a second time and then the hot press
evacuated a third time to a level of 125 mb (95 mm Hg).

The furnace was heated to approximately 500°C and held for 15 minutes. The temperature was then increased at a rate of 600°C/hr. to the maximum temperature of the experiment. The samples were held at temperature for either 1 minute or 60 minutes before the power to the furnace was shut off. The pressure on the material specimens was then released and the specimens allowed to cool with the furnace.

The temperature-pressure schedule employed in these experiments is shown in Table VI-3. The specimens were held at the maximum temperature for 1 hour or 1 minute as indicated.

The three temperature-pressure runs marked with an asterisk were initial runs with only one of the mock extraterrestrial materials in the hot press die (rather than running all three materials together as discussed above). In these experiments a pressure of 6.89 MPa was applied and the temperature was increased at a rate of 600°C/hr. The travel of the hot press ram was continuously monitored in order to determine the temperature at which the mock material began to flow (shrink) in the die. The onset of shrinkage was used as an indication that the solidus temperature of the material had been reached. The formation of a liquid phase above the solidus temperature would promote consolidation of the mock material. The effect of temperature on travel of the hot press ram (and the specimen contained by the graphite die between the upper and lower plungers) is shown in Figure VI-2. The results from these three runs were used to design the remainder of the experiments in the study of the densification behavior of the mock materials during simultaneous application of heat and pressure.

Results and Discussion. The results of the initial three hot press runs with a single specimen in the die are shown in Figure VI-2. Data taken during the hot press run with the mock lunar basalt mixture in the die indicated that expansion of the system occurred up to a temperature of approximately 1080° to 1100°C. At this temperature, shrinkage of the specimen equalled the expansion of the remainder of the load train. Above approximately 1140°C rapid shrinkage of the specimen commenced. The experiment was terminated by shutting off furnace power and releasing the applied pressure when a temperature of 1180°C was achieved.
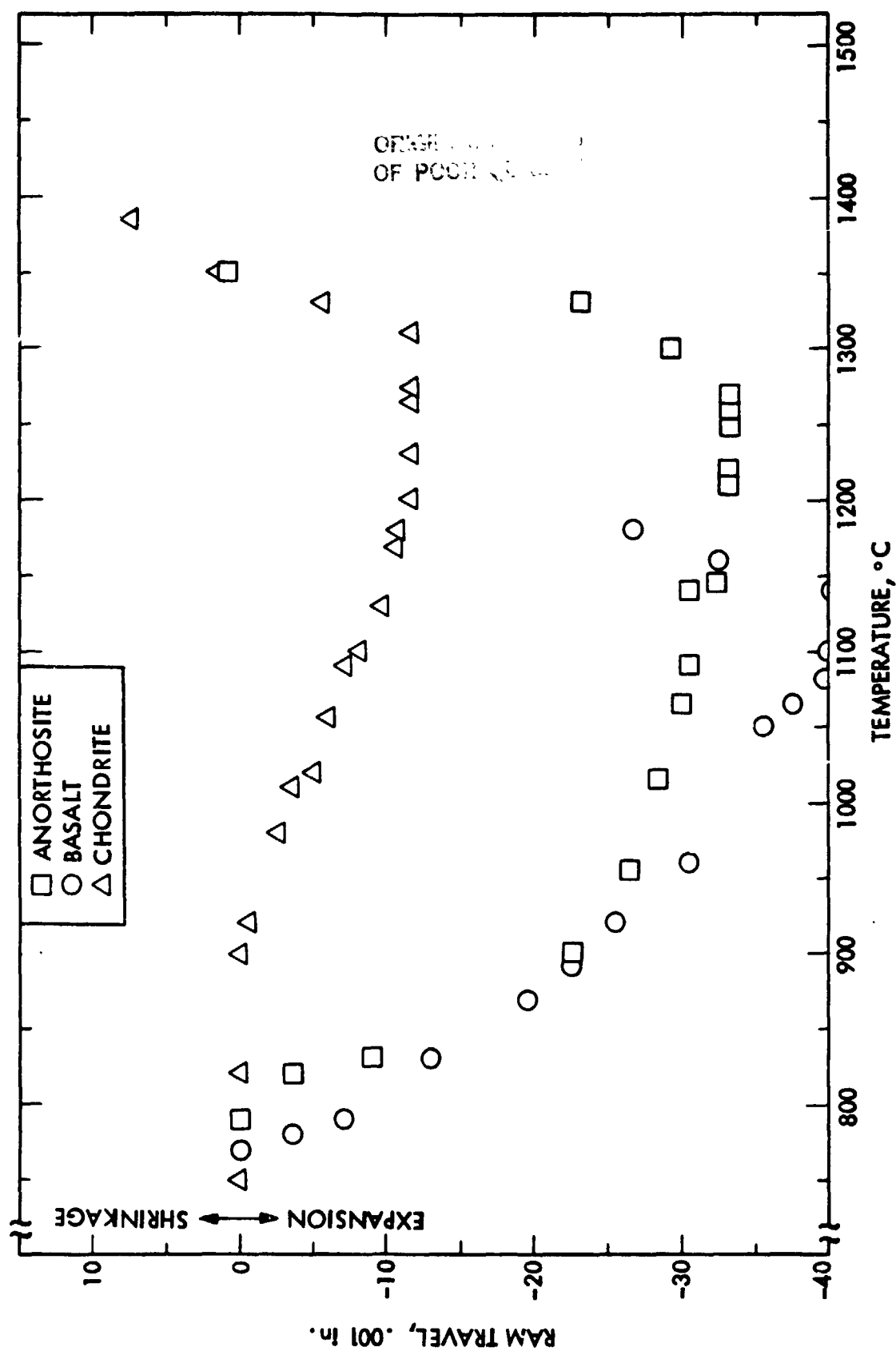
Figure VI-2 Results of Compaction Experiments Under Simultaneous Application of Heat and Pressure

The mock basalt specimen, after removal from the hot press die, was found to have densified and developed appreciable strength. Removal of the surface layer of graphite foil revealed the original grain structure on the specimen surfaces. From examination of the specimen texture it appeared as if some liquid phase sintering occurred during the hot press run. Petrographic examination is planned to determine if microstructural evidence of liquid phase formation can be found.

The data for the hot press run with mock lunar anorthosite mixture (Figure VI-2) showed that the hot press load train expanded as temperature increased up to approximately 1200°C. Above 1200°C the shrinkage of the specimen matched the expansion of the remainder of the load train. Above 1270°C shrinkage of the anorthosite specimen increased rapidly. The experiment was terminated at a temperature of 1350°C. It was interesting that the rate of load train expansion was less than that for the mock basalt mixture at temperatures between 950°C and 1100°C. While the reason for this behavior is not apparent at the present time, it is likely that above 950°C the specimen began to shrink (densify).

The specimen of mock lunar anorthosite had densified to some extent during hot pressing. It could be handled easily and the surface was polished with SiC paper using water to cool the SiC grain and remove the grinding debris from the surface. The grain texture of the original mixture of minerals was evident on the specimen surface.

The data for the hot press run with the mock asteroidal chondrite (Figure VI-2) indicate that shrinkage of the specimen matched the expansion of the remainder of the load train up to approximately 900°C. Expansion of the load train then occurred up to approximately 1200°C. Between 1200° and approximately 1310°C the shrinkage of the anorthosite specimen again equalled the expansion of the remainder of the load train. Above approximately 1310°C shrinkage of the specimen becomes rapid. The experiment was terminated at 1385°C. The overall expansion of the load train containing the chondrite specimen was lower than with either the mock basalt or mock anorthosite materials. This

behavior may have been due to creep of the 20 wt.% metal phase (8% Ni · 92% Fe). However, this hypothesis will have to be confirmed by microstructural evaluation.

Visual examination of the hot pressed chondrite specimen found a dense, strong body with clean, sharp edges. Remnants of the original grain structure were apparent on the surface.

Based on the information obtained from these initial hot pressing experiments and from the differential thermal analysis of the three mock materials, the temperature range of 1050^0 to 1400^0C was selected for the studies on densification of mock extraterrestrial materials by the simultaneous application of heat and pressure.

While the specimens indicated in Table VI-3 have been formed, their evaluation is not yet complete. Final results of these experiments will therefore appear in the next report.

PULLING DEVICE

SHAPE CONTROLLED BY
- DRAWING RATE
- COOLING RATE
- SURFACE TENSION
- VISCOSITY
- G-LEVEL
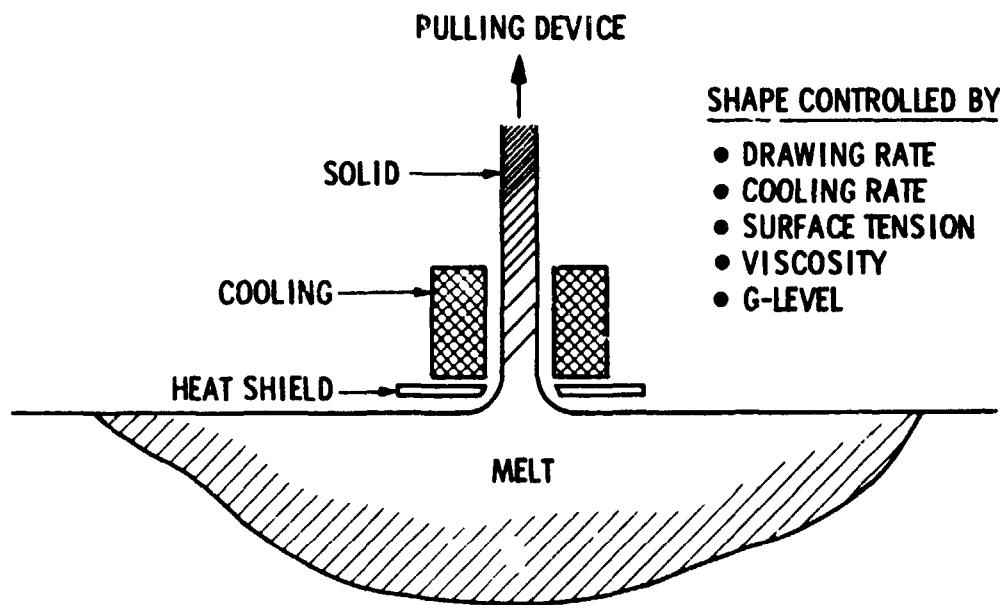
SOLID

COOLING

HEAT SHIELD

MELT

Figure VI-3 Contact-Free Drawing of Silicates from the Melt

2. Production From the Liquid Phase

The extraterrestrial forming of products directly from molten silicates can be performed by pouring molten material into a mold, or by

forcing or pulling molten material through a die or orifice. Under asteroidal microgravity, drawing may even be accomplished without tooling contact, as illustrated in Figure IV-3. The major characteristics of processes being evaluated in this part of the program are presented in Table VI-4.

The raw material in each case is extraterrestrial silicate rock or soil. The process concepts are derived from terrestrial experience in dealing with molten silicate materials. Fusion casting of basalt to form pipe, abrasion resistant parts and chemical resistant parts is a common production method in Eastern Europe (Ref. 72). Fusion casting of refractories for use as glass tank and steel furnace linings is common in the USA and other industrialized countries. Refractory castings with dimensions greater than 2.4m (8 ft.) on a side are commonly produced. Extrusion and drawing are common glass forming methods which may be adapted to the extraterrestrial environment.

Equipment requirements are few and the equipment is extremely simple for forming of products from molten silicates. The silicate raw material may be heated in-situ or transferred to a furnace for melting. In some cases, as in production of transparent glass, the molten material may undergo further treatment in a separate furnace. Means to transfer molten material is required in some situations. Equipment is also required for use in forming the shaped product (similar to molds, extruders, drawing/spinning/blowing apparatus, etc.). It may be necessary to cool the product at a controlled rate in order to allow time to relieve strain set up by differential thermal contraction. Equipment similar to an annealing oven (or insulating "blanket") may thus be a necessary part of the processing operation. Requirements for temperature control can be determined from analysis of terrestrial experiments using mock extraterrestrial materials.

The liquid phase production methods lend themselves to formation of large shapes although a wide range of sizes and shapes can be made. Location of production facilities in a low or microgravity environment would facilitate the production of massive structural members for large space structures. Cast silicates may be utilized in extraterrestrial

Table VI-4 MAJOR CHARACTERISTICS OF LIQUID-STATE SILICATE PROCESSES

	CASTING	EXTRUSION	DRAWING
RAW MATERIAL	SILICATE ROCK AND SOIL →→→		
PROCESS CONCEPT	FUSION CASTING OF BASALTS AND REFRACTORIES	GLASS FORMING →→	(INCLUDES BLOWING AND SPINNING)
PROCESS REQUIREMENTS	• IN-SITU MELTING OR GLASS FURNACE →→→ • MELT TRANSFER EQUIPMENT →→→ (?) • REFRACTORY MOLDS	EXTRUDER	DRAWING/SPINNING/ BLOWING APPARATUS
	• ANNEALING OVENS (?) →→→		
PRODUCTS	LARGE SHAPES, COMPLEX SHAPES	UNIFORM CROSS-SECTION; BEAMS, TUBES, RODS	UNIFORM CROSS-SECTION; FIBERS, BEAMS, TUBES, RODS, BUBBLES
ADVANTAGES	SIMPLICITY, ADAPTABLE TO AUTOMATION, CONTINUOUS, LOW ENERGY REQUIREMENTS, PRODUCE LARGE NUMBERS OF PARTS →→→		
DISADVANTAGES	OBTAINING HOMOGENEOUS MELT, TEMPERATURE CONTROL AND UNIFORMITY →→→		HANDLING OF FIBERS, USE OF GASES (BLOWING)

environments in ways that concrete is used on Earth. Roads may be cast by pouring molten silicates or by melting in-situ. Habitats may be constructed by pouring liquid silicates into forms, by extruding large sections and by drawing or blowing large shapes. Radiation shielding and shielding from particle impact may also be easily formed by liquid phase production methods.

Advantages in utilization of these liquid phase production methods are in general the same. They are simple to perform, can be adapted to automated processing techniques, can be continuous, have low energy requirements and can be used to produce large numbers of parts with a wide range of sizes and shapes. For the production of useful components it is necessary to achieve a melt of homogeneous composition* and free of gas inclusons. Also, handling of fibers produced by blowing molten silicates may pose some difficulty in low-gravity environments.

Literature on crystallization behavior of lunar and meteoritic materials (e.g., Ref. 73-77) has been reviewed as a prelude to studies of liquid phase production methods. Prior work on fusion casting of refractories (e.g., Ref. 78) and of basalts (e.g., Ref. 79) has also been evaluated as part of this investigation. Out of this review of prior work has come the design of initial experiments to demonstrate the formation of simple shapes from terrestrial equivalents of extraterrestrial minerals. Further melt studies were performed to demonstrate the role of cooling rate on the microstructure and phase composition of extraterrestrial materials. These studies were designed to provide information on the effect of a wide range of cooling rates.

The objective of the liquid phase production studies are (a) to develop an understanding of the kinetics of crystallization of extraterrestrial silicates, (b) to develop an understanding of the effect of cooling rate on texture and phase composition of extraterrestrial silicates cooled from the molten state, and (c) to evaluate the properties of extraterrestrial materials produced from the molten state.

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* In some cases, such as the production of foundations, roads, large structural members, and foamed glass, this would not be a consideration.

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a.   Initial Experiments.

Initial studies of liquid phase production methods began with an evaluation of the melting and solidification behavior of terrestrial minerals which are terrestrial equivalents of constituents of extraterrestrial soil.   Three different minerals were heated in an oxygen-natural gas flame and their behavior observed.   These minerals were oligoclase (NaCaAl silicate); olivine (MgFe silicate); and serpentine (hydrated Mg silicate).   The purpose of these studies was to obtain an initial assessment of (a) the behavior of silicates in their liquid state and (b) shapes which could be produced upon cooling these silicates from the melt.
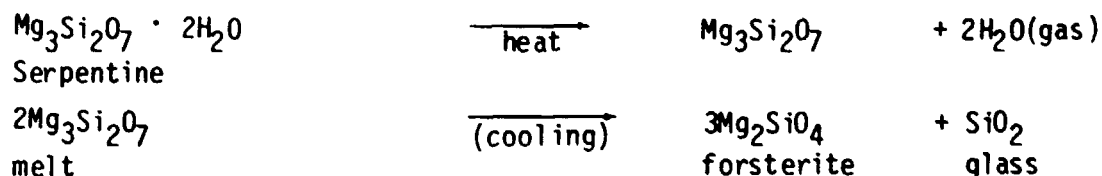
The oligoclase melted in the flame (liquidus temperature approximately 1200^0C) and was blown into bubbles using conventional glass blowing techniques.   These bubbles cracked due to internal strain developed during cooling.   The bubbles formed were translucent and "glassy" in appearance.   X-ray diffraction analysis of both the starting material and the bubbles showed that the oligoclase, crystalline before heating, was a glass (amorphous) after cooling from the melt.   Another sample of oligoclase rock was heated in a graphite resistance heating furnace to approximately 1500^0C and held for 30 minutes at this temperature.   The molten silicate was cooled in the furnace when the power was shut off.   The molten, furnace-cooled sample was analyzed by x-ray diffraction and also found to be amorphous.   This experiment demonstrates that oligoclase can be cooled from the melt at relatively slow rates ($\sim 1^0$C/sec during the first several hundred seconds) and still remain in the glassy state.

Additional experiments demonstrated that oligoclase can be drawn into fibers.   These fibers, drawn manually from a molten mass of material in an oxygen-natural gas flame were approximately 0.3m (12 in.) in length and 500 μm (0.02 in.) in diameter.

Olivine was also melted in an oxygen-natural gas flame.   The viscosity of the melt appeared to be similar to that of water.   Because of the low viscosity it was not possible to blow bubbles or draw fibers

under terrestrial gravity conditions. X-ray diffraction analysis of the
crystalline phases present in the starting material and in the material
cooled from the melt was performed. It was found that the material
cooled from the melt was crystalline and had the same phase composition
as the starting material. The x-ray diffraction analysis also indicated
that the olivine used in this experiment was magnesium-rich, i.e., closer
to the composition of forsterite (Mg_2SiO_4) than to fayalite
(Fe_2SiO_4). The olivine was also heated to $1600^\circ C$ in a differential
thermal analysis (DTA) cell without observing melting of the sample. The
liquidus temperature of the olivine sample used in this study is thus
above $1600^\circ C$ (the temperature limit of the DTA unit).

Serpentine, a minor constituent of carbonaceous chondrites (~1%),
was melted in the oxygen-natural gas flame. The melt appeared to have a
viscosity similar to that of water. A large pore, present at the surface
of the solidified material, was formed by gas coming from the serpentine
crystal structure. The presence of forsterite and an amorphous phase in
the material formed from the melt was determined via x-ray diffraction
analysis. The occurrence of these two phases can be explained by the
following reaction sequence:

$$Mg_3Si_2O_7 \cdot 2H_2O \xrightarrow{\text{heat}} Mg_3Si_2O_7 + 2H_2O(gas)$$
Serpentine

$$2Mg_3Si_2O_7 \xrightarrow{\text{(cooling)}} 3Mg_2SiO_4 + SiO_2$$
melt $\qquad\qquad\qquad\qquad\qquad$ forsterite $\quad$ glass

The chemically bound water comes out of the structure as a gas. The
molten $Mg_3Si_2O_7$ crystallizes on cooling to form forsterite
(Mg_2SiO_4) and a glass phase (SiO_2).

Of the three terrestrial equivalents of extraterrestrial minerals
studied, oligoclase was the only one which behaved such that glass
forming techniques could be utilized in producing shapes from the melt.
However, other methods of forming shapes from a melt of low viscosity
exist. A notable example is the EFG method developed by Mobil-Tyco, Inc.
which is being applied to the drawing of rods, tubes and ribbons of
Al_2O_3 (melting point ~$2050^\circ C$) and of Si ribbon (melting point

1410°C). The EFG method of forming products from the melt as well as other methods will be evaluated in a subsequent experimental program.

b. Melt Studies

Experimental Procedure. The three mock extraterrestrial materials described in Section III-F were utilized in this study. These mock materials represent typical compositions which might be found in the lunar highlands (anorthosite), lunar maria (basalt) and on some asteroids (chondrite). The purpose of these studies was to evaluate the effect of cooling rate on the crystallization behavior of selected extraterrestrial silicates.

Differential thermal analysis (DTA) of each of the mock materials mixtures was performed to determine reactions and significant phase changes, including the onset of melting (solidus temperature). Specimens were heated in an argon atmosphere at a rate of 10°C/min. from 25° to 1400°C. The temperature of the mock materials was subtracted from that of an inert reference material and this temperature difference was plotted versus the temperature of the reference material. Alpha alumina was selected as the inert reference material. In addition, the reference material was run against itself to establish a baseline.

The furnace* used to melt the mock materials has a water-cooled stainless steel shell and employs a tungsten mesh heating element. The furnace can be operated with a vacuum, inert or reducing environment. It is capable of attaining temperatures above 2000°C over a 5 cm diameter and 10 cm high hot zone. The furnace temperature was controlled by monitoring the sample temperature via an optical pyrometer and by manually adjusting the power to the heating element as necessary.

For the initial run three molybdenum crucibles, each containing one of the three mock materials, were placed on an alumina setter plate. The setter plate was placed on the tungsten hearth of the furnace. After the

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*   Astro Industries, Inc., Model 1100V 3580W1.

first run a tantalum foil boat was placed between the crucibles and alumina setter. The Ta foil boat and alumina plate provided protection to the furnace from the materials being melted should they flow out of the crucibles. After placing the samples in the furnace the furnace was evacuated to below 4×10^{-4} torr and backfilled with helium. During the furnace runs a flow of helium of 16 cm^3/sec was maintained. The He was exhausted from the furnace through a container of silicone oil in order to prevent back-diffusion of oxygen into the furnace.

Quenching of the specimens was accomplished by turning off power to the furnace while increasing the flow of helium. A W-WRe thermocouple used to monitor temperature was located at the midpoint of the furnace hot zone and adjacent to the crucibles containing the mock extraterrestrial materials. Slower cooling rates used in this study were accomplished by decreasing power to the furnace while monitoring the rate of temperature decrease.

The mock materials were contained in molybdenum crucibles 1.27 cm inside diameter and 1.90 cm high. Molybdenum lids were used to create a closed system for the mock material during the furnace runs.

For the initial experiments in the study of the effect of cooling rate on the crystallization behavior of the mock extraterrestrial materials a rapid quench and two slow cooling rates were selected. The experiments to determine the effect of annealing materials after cooling through their solidus temperatures were postponed until the data from the crystallization experiments could be analyzed. The initial cooling rates selected were approximately 1^0C/min., 10^0C/min. and a rapid quench. An analysis of the experimental results showed the actual rates to be 0.8^0C/min., 7^0C/min. and 500^0C/min. Data was also available from one run made at a cooling rate of 4^0C/min.

The mock extraterrestrial materials were first heated to a temperature above their liquidus and held for one hour before cooling commenced. The first set of three materials was heated to 1400°C, held for one hour and quenched by shutting off power and flowing He through the furnace. The quench rate, monitored by a W-WRe thermocouple, was approximately 500°C/min.

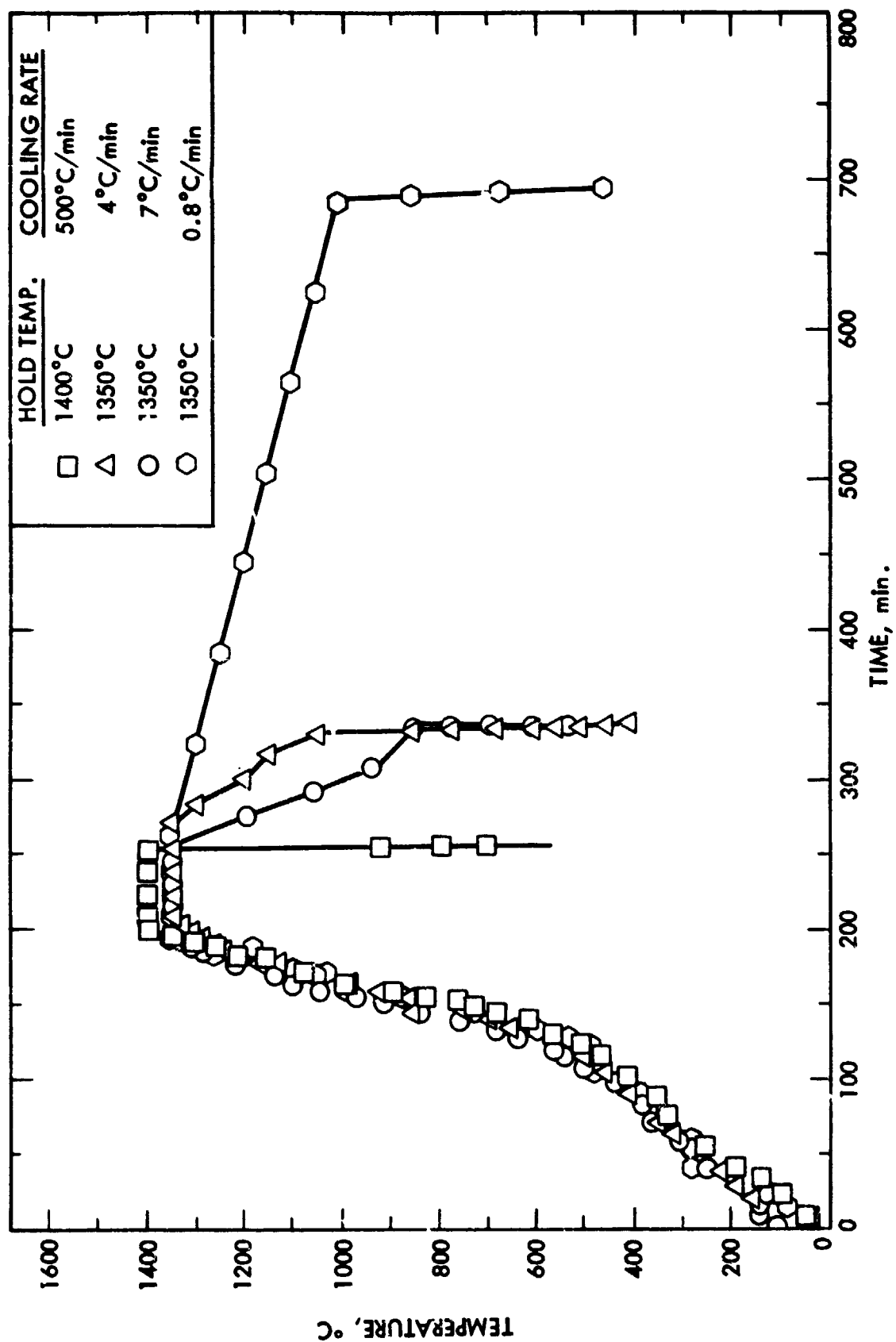Figure VI-4 Thermal History of Specimens Formed in Melt Studies

	HOLD TEMP.	COOLING RATE
□	1400°C	500°C/min
△	1350°C	4°C/min
○	1350°C	7°C/min
⬠	1350°C	0.8°C/min

Upon examination of the specimens after their removal from the
furnace it was observed that the basalt had flowed from its crucible,
around the crucible lid and onto the alumina plate. The material on the
alumina plate was "glassy" in appearance and exhibited a conchoidal
fracture mode. Because of this overflow problem it was decided to lower
the maximum temperature to 1350°C.

For the remainder of the experiments the specimens were heated to
1350°C and held for 1 hour before cooling commenced. The heating and
cooling curves for the four (4) sets of specimens are shown in
Figure VI-4. The curves are linear above 1050°C for the 500°C/min.,
7°C/min. and 0.8°C/min. cooling rates and approximately linear for
the sample cooled at 4°C/min. After cooling, the crucibles containing
the specimens were sectioned along their axes with a diamond wheel, as
illustrated in Figure VI-5. The center portion of each sample was used
to make a thin section for petrographic analysis while the outside
portions were used for preparing a polished section and for phase
identification by x-ray diffraction.

Figure VI-5 Crucible Dimensions and
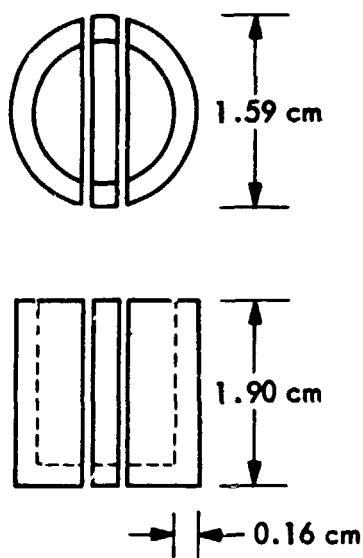Sectioning Pattern Used in Melt Studies

Results - Differential Thermal Analysis. Data from the initial
differential thermal analysis (DTA) of the three mock materials are
illustrated in Figure VI-6. This figure shows the difference in
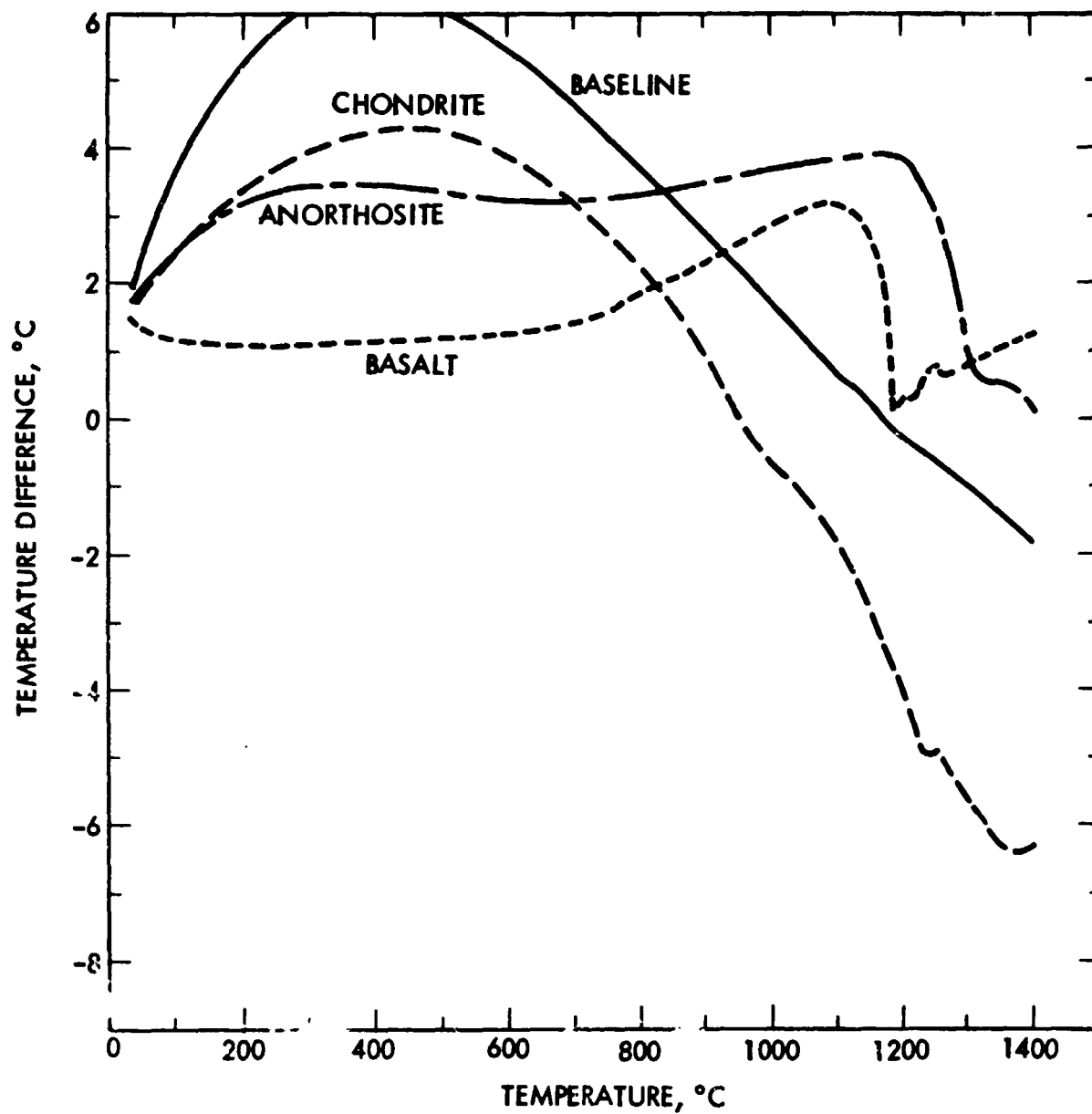temperature between the mock materials and the inert reference material,

Figure VI-6 Differential Thermal Analysis of Three Mock
Extraterrestrial Materials

ΔT, versus the temperature of the reference material. Also in Figure VI-6 is a curve showing the data obtained from a run of the standard material, alpha alumina against itself. Such a baseline run shows the equipment error inherent in such experiments. If there were no sources of error in the DTA equipment (or if the sum of the errors was zero) the baseline curve would be horizontal and indicate a zero temperature difference. The actual value of ΔT as a function of temperature for runs with the mock materials can be found by subtracting the temperature difference of the baseline curve from that of the curve obtained with the mock materials. The actual ΔT obtained in this manner for each of the mock materials is shown in Figure VI-7.

The results of these initial runs, illustrated in Figure VI-7, show a broad endotherm for all three of the mock extraterrestrial material mixtures at temperatures below 800°C. The low temperature portion of the curves may be controlled by a difference in heat capacity between the mock material mixtures and the alpha alumina reference material.

Above 900°C the temperature of mock chondrite begins to lag behind that of the reference material by an ever increasing amount. This behavior is indicative of an endothermic reaction, as yet unidentified, which occurs over a broad temperature range.

The mock basalt and anorthosite show behavior opposite to that of the chondrite above 900°C. The temperature of the basalt and the anorthosite actually leads that of the inert reference material as if there were an exothermic reaction taking place. As the temperature of these two materials is increased above 900°C there is a change in slope of the curves from positive to negative sign. The temperature at which the curves begin their change in sign may be the temperature at which the onset of melting occurs. If this is true, the solidus temperature can be found from Figure VI-7 to be approximately 1200°C for the anorthosite and approximately 1070°C for the basalt.

Additional studies of the behavior of these mock extraterrestrial material mixtures are required in order to ascertain the cause of the behavior illustrated in Figure VI-7. Additional baseline calibration runs are planned as well as experiments with the mock material mixtures and with their individual components.
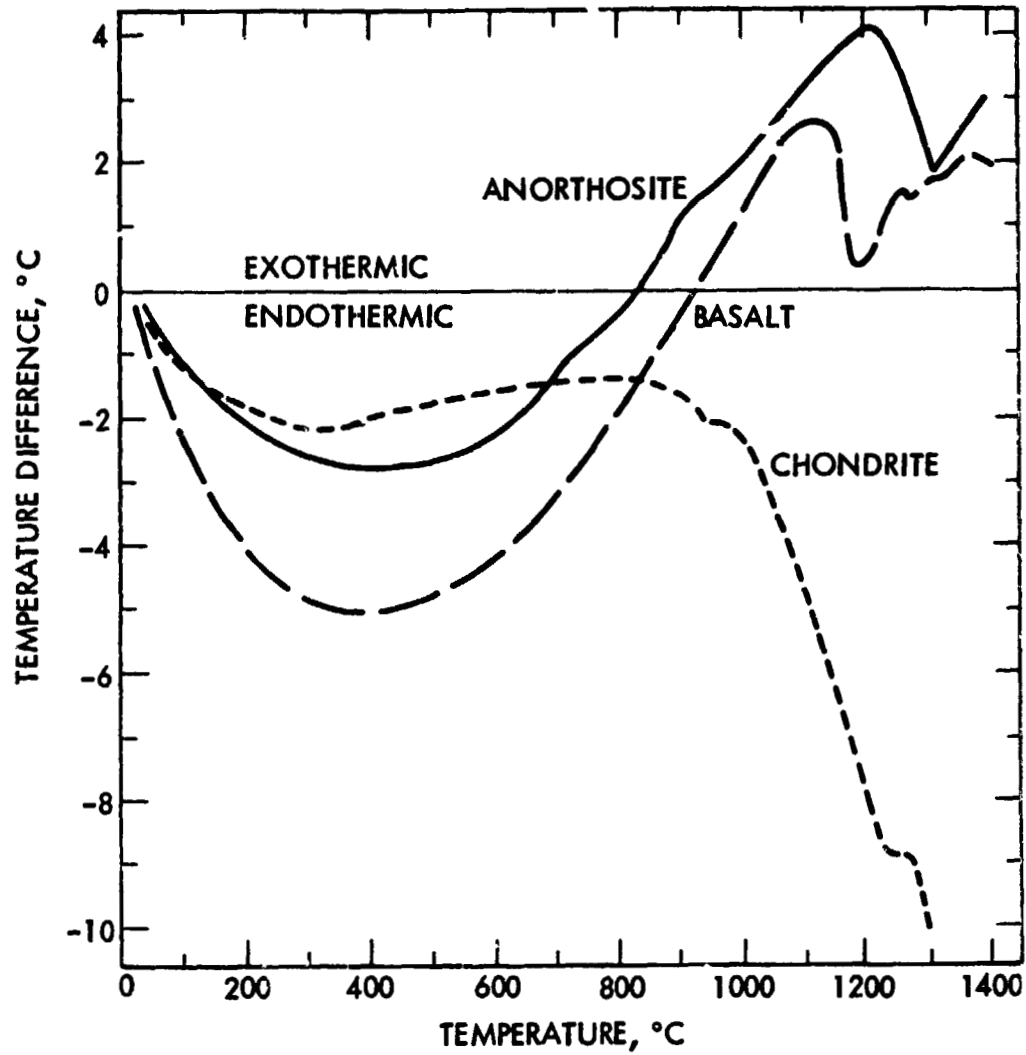
Figure VI-7 Differential Thermal Analysis After
Accounting for Shift in Baseline

X-ray Diffraction Analysis. Samples of each of the starting
mixtures of the mock extraterrestrial materials and samples of the
materials formed by cooling from 1350°C and 1400°C were analyzed by
conventional x-ray powder diffraction techniques. The results are shown
in Table VI-5. In the following evaluation of the effects of cooling
rate, the x-ray diffraction (XRD) patterns of the mock materials formed
from the melt are compared with those of the starting materials. Where
the XRD patterns of the starting minerals are best indexed to a different
mineral in the ASTM x-ray powder diffraction file, the ASTM designation
is listed in parenthesis.

Table VI-5 X-RAY POWDER DIFFRACTION ANALYSIS OF MOCK MATERIALS

TREATMENT	ANORTHOSITE	BASALT	CHONDRITE
Starting Mixture	bytownite (oligoclase) olivine (forsterite)	augite bytownite (oligoclase) ilmenite	olivine (forsterite) iron bronzite (enstatite) oligoclase
Held 1350°C, 1 hr, cooled 0.8°C/min.	analbite olivine (forsterite) unknowns	augite unknowns	olivine (forsterite) unknowns
Held 1350°C, 1 hr, cooled 4°C/min.	analbite olivine (forsterite) unknowns	augite peaks partially amorphous	olivine (forsterite) spinel phase? unknowns
Held 1350°C, 1 hr, cooled 7°C/min.	analbite unknowns	augite unknowns	olivine (forsterite) bronzite (enstatite), trace unknowns
Held 1400°C, 1 hr, cooled 500°C/min.	amorphous	augite unknowns	olivine (forsterite) unknowns

<u>Anorthosite</u>. The starting mixture of mock lunar anorthosite was prepared from bytownite, olivine and augite. The x-ray pattern for this mixture indexed (a) closer to oligoclase than bytownite and (b) closer to forsterite than olivine. Augite, present at the 1% level in the starting mixture, did not appear in the x-ray pattern. The d-spacings for the oligoclase structure found in the ASTM card file matched the XRD pattern of the mock lunar anorthosite better than did the catalogue value for bytownite. The oligoclase had the composition 0.83 $NaAlSi_2O_8$ · 0.16 $CaAl_2Si_2O_8$ while the bytownite was lower in Na and higher in Ca with the chemical formula 0.23 $NaAlSi_2O_8$ · 0.77 $CaAl_2Si_2O_8$.

The olivine used in the starting mixture of anorthosite had an XRD pattern corresponding more closely to forsterite (Mg_2SiO_4) than to olivine ($2(Fe_{0.94}, Mg_{0.06})SiO_4$). However, the olivine used in the present study was expected to have an iron/magnesium ratio much less than the 15.7 (0.94/0.06) of the ASTM XRD standard and thus be closer to forsterite in composition.

The anorthosite was cooled at $0.8^\circ C$ per minute from $1350^\circ C$. The crystalline phases observed in XRD analysis of the solidified material were analbite, the high temperature form of albite ($NaAlSi_3O_8$), and olivine (forsterite). In addition there were a number of diffraction lines which could not be identified.

The anorthosite cooled from $1350^\circ C$ at $4^\circ C/min$. was below the approximate liquidus temperature of $1200^\circ C$ in about 38 minutes. The crystalline phases present in the material cooled from the melt were the same as observed in the material cooled at $0.8^\circ C/min$., i.e., analbite and olivine. In addition there were a number of diffraction lines which could not be identified.

On cooling the anorthosite from 1350°C at approximately 7°C/min. only one phase which could be identified was formed. That phase was analbite. There were also several diffraction lines which could not be indexed. No olivine (forsterite) was observed in this specimen.

The anorthosite sample held at 1400°C for 1 hour and quenched at 500°C/min. was found to be amorphous. No crystalline phases were detected by XRD.

The occurrence of analbite as a result of cooling molten material originally containing bytownite (oligoclase) can be explained by use of the phase diagram in Figure VI-8 (Ref. 80). This is a binary phase diagram for albite ($NaAlSi_3O_8$) and anorthite ($CaAl_2Si_2O_8$). This diagram shows that compositions in the oligoclase region of the diagram (indicated by the hatched area) first form analbite on cooling from 1350°C.
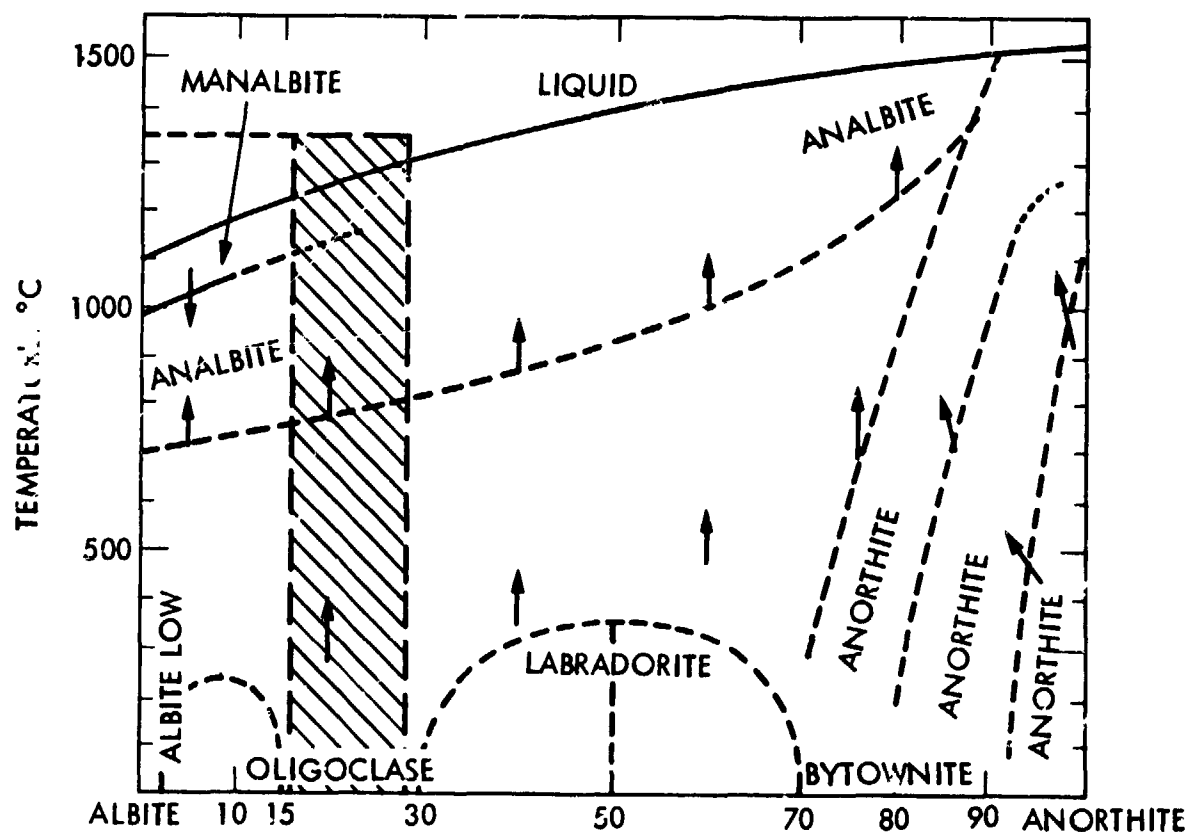

Figure VI-8 Phase Diagram of Albite-Anorthite (from Ref. 80). The shaded area represents the temperature-composition field of oligoclase

Olivine (forsterite) precipitated from the melt on cooling at 0.8°C/min. and 4°C/min. However, at quench rates of 7°C/min. and above forsterite (olivine) did not have sufficient time to precipitate from solution in sufficient quantity to produce a recognizable XRD pattern.

Basalt. The mixture of minerals making up the mock lunar basalt had an XRD pattern which corresponded to augite, bytownite (oligoclase) and ilmenite. Olivine, present at the 3 weight percent level, could not be detected.

The mock lunar basalt was cooled from 1350°C at approximately 0.8°C/min. XRD analysis showed the presence of augite along with low intensity diffraction lines which could not be identified. The presence of bytownite (oligoclase) and ilmenite, present in the starting mixture, was not observed.

The material cooled from 1350°C at approximately 4°C/min. was found to be partially amorphous and to contain augite. As in the sample cooled at 0.8°C/min., augite (which was the major constituent (50 wt.%) of the starting mixture) was the only phase detected.

The material cooled from 1350°C at 7°C/min. contained augite and minor phases which produced diffraction lines which could not be identified. The material cooled from 1400°C at ~500°C/min. had a diffraction pattern similar to that of the material cooled at 7°C/min. Augite was the major phase present. There were additional diffraction lines present which could not be identified.

The unidentified diffraction lines were all of low intensity ($I/I_0 <$ 15%) and are likely a consequence of the formation of a solid solution of $Na(Ca, Mg, Fe)Al(Ti, Si)O_9$, perhaps contained in a glass matrix. Petrographic analyses of thin sections and of polished sections of the mock basalt cooled at the four (4) rates discussed above are to be performed during the next reporting period.

Chondrite. The mixture of minerals making up the mock chondrite was formulated to simulate the composition of a typical ordinary chondrite of asteroidal (meteoritic) origin. This mixture was the most complex of the three mock extraterrestrial materials prepared. There were six different constituents in the mixture: olivine, bronzite, Ni-Fe, oligoclase, troilite and apatite. The first four constituents were present in quantities between 10 and 33 wt.% while troilite and apatite were present at the 5 and 4 wt.% level, respectively.

The XRD analysis of the starting mixture of mock chondrite showed the presence of olivine (forsterite), bronzite (enstatite, $MgSiO_3$), α-iron and oligoclase. Troilite and apatite were not detected.

The material heated to 1350°C, held for 1 hour and cooled at approximately 0.8°C/min. was found to contain olivine (forsterite). No other phases were detected via XRD. The XRD peaks obtained with this material were of low intensity and broadened compared to the XRD pattern of the starting mixture. This is an indication that the sample may be only partially crystalline and/or have a disordered structure.

The material held at 1350°C for 1 hour and cooled at 4°C/min. was found to contain olivine (forsterite). There was also a strong diffraction line at a d-spacing of 2.526 Å which could not be specifically attributed with any known phase. However, it is likely that this line is due to the presence of a spinel phase (not magnetite). The peaks in the XRD pattern for this material were sharp and strong indicating a high degree of crystallinity.

The material held at 1350°C for 1 hour and cooled at 7°C/min. was found to contain olivine (forsterite) and a trace amount of bronzite (enstatite). The major peak at 1.634 Å in this diffraction pattern was not identified.

An XRD analysis of the material heated to 1400°C and held for 1 hour before quenching at approximately 500°C/min. showed the presence of olivine (forsterite) as the major phase. There were several minor peaks in the XRD pattern which could be not identified.

C-2

Olivine was the major phase in each of the specimens cooled from the melt. The presence of a metallic phase (the initial mixture contained 20 wt.% of Ni-Fe metal) was not observed in any of the XRD analyses. The presence of unidentified diffraction peaks of low intensity ($I/I_0 < 15\%$) may be an indication of a solid solution of the chondrite material mixture in the melted and quenched specimens. Petrographic analyses of thin sections and of polished sections of the quenched mock chondrite specimens are to be performed during the next reporting period.

c. Thermo-Physical Model Studies.

An estimate of the rate of crystallization of a lunar basalt formed by casting into an open mold on the lunar surface is presented. This information provides an estimate of the time required to form large structural elements by casting from a melt of lunar material.

Calculation of Solidification Rate of Lunar Basalt. An estimate of the rate of solidification of an extraterrestrial material was made in order to determine the time required to cool cast silicate structures of various sizes. The rate of cooling of a cast lunar basalt from its liquidus to its solidus temperature was determined. Calculations of the effect of size on the rate of cooling from the solidus to temperatures where the structures can be handled ($\sim 500^0$-600^0C) and below are in progress. Cooling rates as a function of size for drawn and extruded extraterrestrial silicate structures will be reported at the end of FY'82.

The time of solidification of a lunar basalt cast into a mold was calculated based on the model established by Provost and Bottinga which was confirmed by measurements on volcanic lava flows (Ref. 81). It was assumed that the casting would be performed on the lunar surface. Heat loss would be through conduction into the lunar surface and through radiation into space. The time required for complete crystallization in cooling from the liquidus through the solidus tempe ature was determined as a function of the thickness of the casting from the following relationship

$$t = 0.0123d^{1.943}$$

where t is the time in hours and d is the thickness of the casting in centimeters. The solidus and liquidus temperatures are taken as 1100°C and 1200°C, respectively. These are average values of data taken from the literature (Ref. 81).

The relationship between the time for complete crystallization in cooling from 1200°C to 1100°C and the thickness of the solid casting as calculated from the above equation is shown in Figure VI-9.

For example, a casting 30.48 cm (1 foot) thick takes 9.4 hours to cool to the solidus temperature. It would take considerably longer to cool to a point (~500°C) where it could be handled by mechanical equipment. The rate of cooling goes as approximately the square of the thickness. Since conductive heat transfer occurs with a similar time dependence ($t \propto \ell^2/k$ where t is time, ℓ is a characteristic length and k is thermal diffusivity) it is hypothesized that the rate of heat loss is controlled by the rate of conduction of heat from the interior of the material to the surfaces.

Future Work. A numerical model of the casting process is being developed. This model will provide information on the temperature distribution in a cast slab as a function of time. The rate of cooling of the casting will be established as a function of material properties over a temperature range from above the liquidus temperature to below the solidus temperature.

d.   Other Liquid-Phase Processing Studies (planned)

(1) Casting Studies. The initial casting studies are designed to demonstrate the feasibility of casting molten silicates of extraterrestrial composition into coherent shapes (i.e., with no cracks or other large defects). The cooling rate will be controlled at a level determined in the melt studies, described above. Density, specific gravity, and strength will be measured. X-ray diffraction will be used for crystalline phase identification and optical microscopy used to evaluate crystalline texture and to confirm the XRD analysis. This work will begin in FY'82.
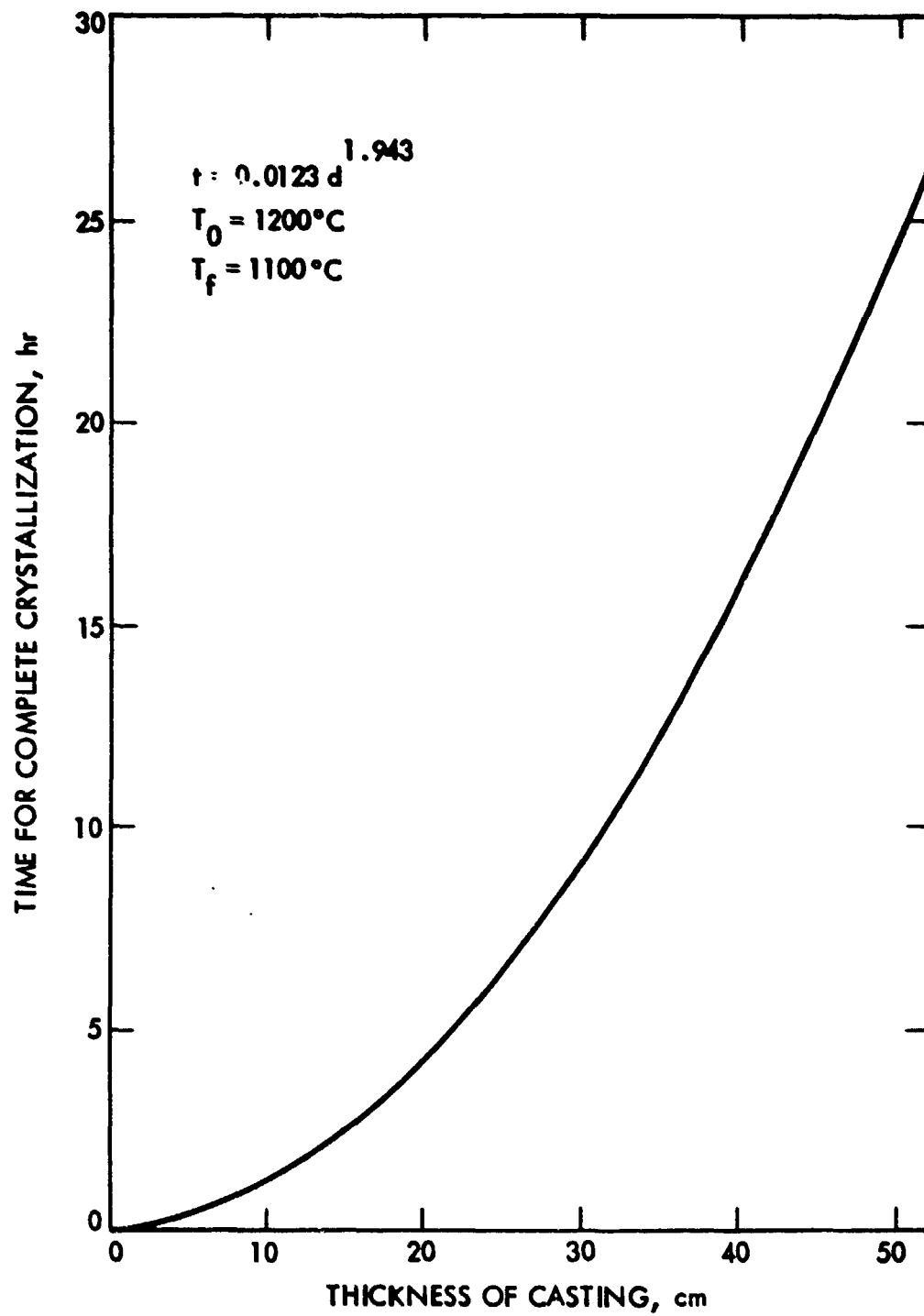
Figure VI-9   Crystallization Time of Cast Lunar Basalt

(2) __Drawing Studies__. The experimental portion of this study is scheduled for FY'83. Initial experiments with terrestrial materials have been described above. A determination of the drawing rates which are possible in low and near-zero gravity environments will be made. Equipment costs for setting up laboratory experiments are to be determined and a decision made as to the purchase of this equipment. If the decision is affirmative, then equipment will be purchased and made operational in FY'83 with drawing experiments (1g) scheduled before the end of the fiscal year.

(3) __Extrusion Studies__. Basic equipment to perform extrusion of molten silicates is available in the JPL Materials Laboratory and can be utilized after some modifications. The modifications and associated costs will be determined and a decision made as to whether to proceed with the modifications. If so, equipment modification and debugging will be carried out in FY'83. The range of extrusion rates theoretically possible will be calculated. Extrusion experiments will be carried out in FY'84 if a decision is made to pursue these experiments.

(4) __Melt-Extraction Studies__. This initial study is designed to provide information on the segregation of phases in partial melts and fully liquid melts. Sample preparation will be performed jointly with that for the Particle Compaction Project. Melt-freeze experiments are to be performed in order to determine the extent of phase segragatic in a 1g environment. The extent of segregation will be determined by x-ray diffraction and optical microscopy. If the results warrant further investigation a decision will be made to either do larger-scale work in-house or to contract the work to a university. The results of this study will be incorporated in the FY'82 year-end report.

(5) __Gas-Evolution Studies__. This study is designed to determine the chemical species evolved during heating of silicates of lunar and asteroidal origin and to determine the temperature dependence of dissociation. Particular attention will be paid to evolution of H_2, H_2O, OH and the metallic elements such as Fe,

Ni, Ti, Ca and Si which are of interest in space construction. The literature is being reviewed to determine if this information is available from earlier studies. The analysis can be performed by use of a mass spectrometer attached to a Knudsen cell. While there are a number of mass spectrometers at JPL there are no Knudsen cells. If the information required is not av.ilable in the literature a decision will be made to either buy the required equipment and have the work performed in-house or to contract the work outside JPL. The results of this phase of the project will be included in the FY'82 year-end report.

B. EXTRACTION OF METALS AND OXYGEN FROM SILICATES

Section B discusses chemical, electrolysis and vapor-phase reduction processes for the extraction of metals and oxygen from silicates. Electrolysis and vapor-phase reduction have been found most promising, as they are highly adaptable to the material and environmental resources of space and probably can be developed to require almost no supply of consumables from earth.

In contrast, chemical processes represent a typical terrestrial technology whose transfer into space is considered impractical in view of their inherent complexity in facilities and operations, and in view of the extensive import requirements of - often hazardous - chemicals from earth. They further do not lend themselves to the production of oxygen. For these reasons, chemical processes have not been actively pursued except for a literature survey. A few examples as suggested in the literature (Ref. 27, 34, 35) are included in the first part of this section merely for completeness.

1. Chemical Processes

A general description of chemical processes is not possible, since each process applies to the production of one specific metal only. The following discussion is, therefore, confined to selected processes for the production of aluminum, iron and titanium (Ref. 34) as they are of prime interest with regard to raw material resources and product usefulness.

a. Aluminum

The major source for extraterrestrial Al is "plagioclase" concentrate which contains anorthite, $(Ca, Na) (Al, Si)_4 O_8$. The soda-lime sintering process of the Bureau of Mines proceeds by the following reaction:
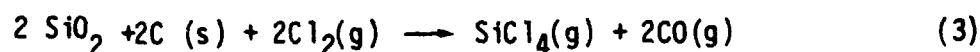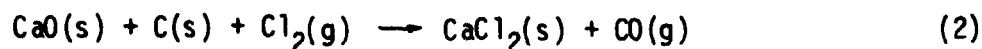
$$CaAl_2Si_2O_8(s) + 3CaCO_3(s) + Na_2CO_3(s) \longrightarrow$$
$$\longrightarrow 2NaAlO_2(s) + 2Ca_2SiO_4(s) + 4CO_2(g)$$
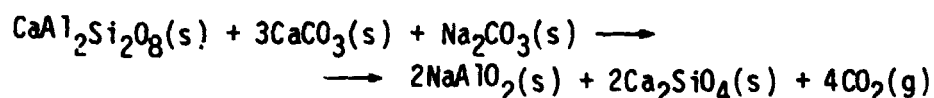
The reaction products are treated to obtain alumina (Al_2O_3). The soda lime sintering process requires the use of large amounts of Ca and Na carbonates, not available on the moon.

More suitable processes for treating anorthite have to be considered such as:

(1) Carbothermic reduction, which will not be discussed here due to some anticipated extensive problems.

(2) Carbochlorination process, followed by electrolysis of the chlorides can be performed in two steps:

Step 1. Carbochlorination Process

The low temperature carbochlorination process with Cl_2 is preferred over the high temperature process (in the latter, corrosion and other problems arise). $AlCl_3$ will be used to extract Al by electrolysis as will be shown in Step 2. The following reaction equilibria are to be considered in the thermodynamic analysis of the process:

$$Al_2O_3(s) + 3C(s) + 3Cl_2(g) \longrightarrow 2AlCl_3(g) + 3CO(g) \qquad (1)$$

$$CaO(s) + C(s) + Cl_2(g) \longrightarrow CaCl_2(s) + CO(g) \qquad (2)$$

$$2 SiO_2 + 2C (s) + 2Cl_2(g) \longrightarrow SiCl_4(g) + 2CO(g) \qquad (3)$$

The net reaction is:

$$CaO \cdot Al_2O_3 \cdot 2SiO_2(s) + 8C(s) + 8Cl_2(g) \longrightarrow$$

$$\longrightarrow CaCl_2(s) + 2AlCl_3(g) + 2SiCl_4(g) + 8CO(g) \tag{4}$$
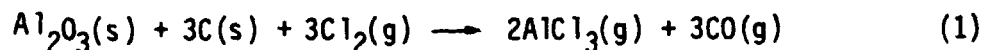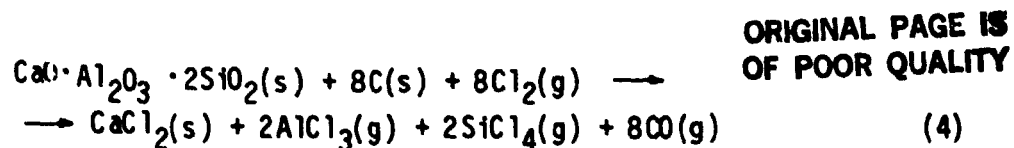
The optimum temperature for anorthite carbochlorination is 675-770°C.
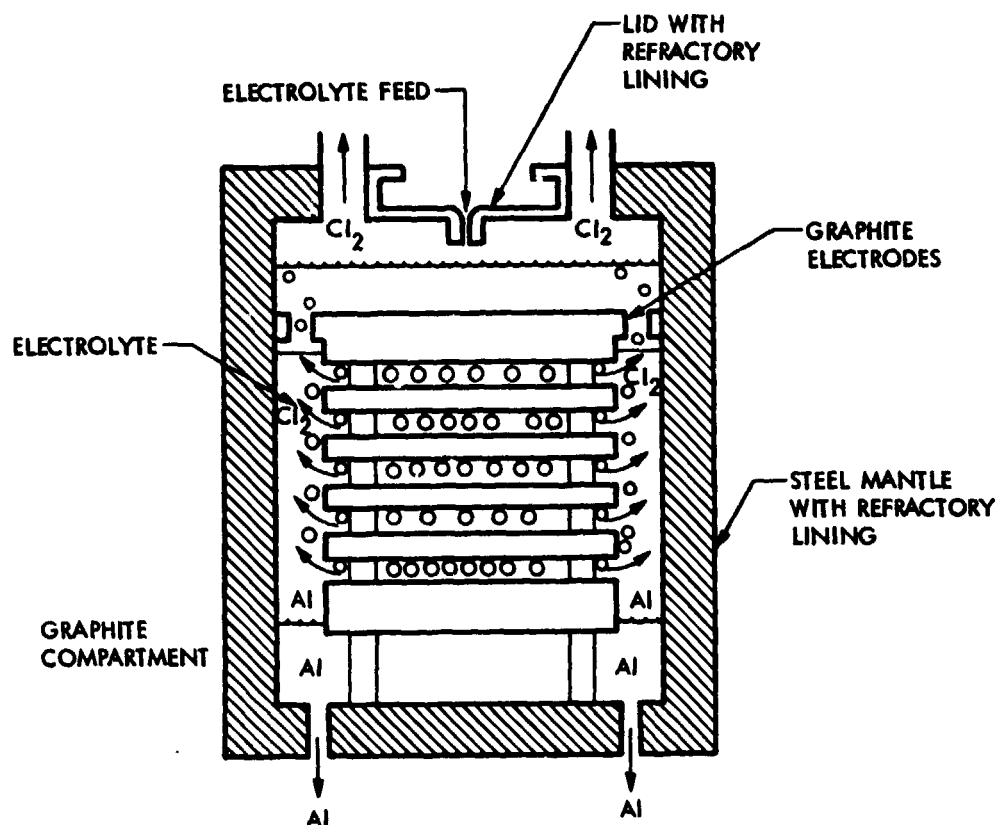
Figure VI-10 Bipolar Cell for Electrolysis of Aluminum Chloride (Alcoa)

Step 2. Alcoa Electrolysis Process:

Al is obtained by the electrolysis of $AlCl_3$ dissolved in a mixture of alkali or alkaline earth chlorides. The electrolysis is performed under inert conditions in a sealed cell consisting of bipolar carbon electrodes. Each bipolar electrode behaves as a cathode on its top surface and as an anode on its bottom surface. During operation all electrodes remain immersed in the electrolyte at ~700°C. A current is passed between the electrodes. The energy consumption of the cell is 9 KWhr/kg of Al produced. A useful feature of the cell, which is shown in Figure VI-10, is that the Cl_2 generated at the anode sweeps the Al away from the cathodes and enhances the coalescence of Al droplets. The pumping effect of the Cl_2 bubbles maintains a continuous

electrolyte flow across the cell, thus preventing the formation of Al pools on the electrodes. Chlorine collects at the top of the cell, while molten Al falls to the bottom of the cell. Liquid Al is passed through a filter to separate it from any excess electrolyte. The electrolyte is recycled into the carbochlorination step.

The calcium metal can be obtained by processing the CaCl, a by-product of the aluminum extraction from anorthite.
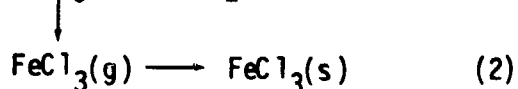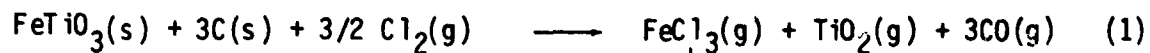
b. Titanium and Iron

The main source for extraterrestrial Ti and Fe is ilmenite, $FeTiO_3$. Several extraction processes have been suggested.

(1) Ilmenite is reduced with coke (coal) or hydrogen at elevated temperatures. The iron is separated from the TiO_2 by leaching with sulfuric acid. The TiO_2 residue is chlorinated in the presence of C, to form $TiCl_4$, which is further reduced by Mg, to yield Ti sponge.

(2) NaOH treatment: Ilmenite is treated with molten NaOH the TiO_2 being preferentially dissolved, leaving Fe_2O_3 which is insoluble in NaOH. The undissolved Fe_2O_3 is separated, and the liquid is leached with hot water. This process has been tested only on a bench scale.

(3) Carbochlorination process: Ilmenite is added to carbon, enough to reduce FeO, and then chlorinated at $800^\circ C$ in a fluidized bed. The Fe is selectively chlorinated yielding $FeCl_3(g)$, while TiO_2 remains in the fluidized bed. The reactions involved in the process are:

$$FeTiO_3(s) + 3C(s) + 3/2\ Cl_2(g) \longrightarrow FeCl_3(g) + TiO_2(g) + 3CO(g) \quad (1)$$

$$\downarrow$$

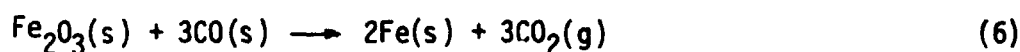$$FeCl_3(g) \longrightarrow FeCl_3(s) \quad (2)$$

Fe and Ti are extracted using the following processes:

(a) Fe extraction: The ferric chloride obtained from the carbochlorination of ilmenite can be reduced directly by H_2 at $\sim 700^\circ C$, as follows:

$$2FeCl_3(s) + 3H_2(g) \longrightarrow 2Fe + 6HCl \qquad (3)$$

Hydrogen chloride (HCl) is a useful by-product and can be used in the chlorination process. Alternatively, $FeCl_3$ can be oxidized in a fluidized bed at 300-500°C to produce Fe_2O_3, which can be reduced with either H_2 or C below 1000°C to obtain low carbon steel or Fe as follows:

$$2FeCl_3(s) + 3/2\ O_2(g) \longrightarrow Fe_2O_3(s) + 3Cl_2(g) \qquad (4)$$

$$Fe_2O_3(s) + 3H_2(g) \longrightarrow 2Fe(s) + 3H_2O(g) \qquad (5)$$

$$Fe_2O_3(s) + 3CO(s) \longrightarrow 2Fe(s) + 3CO_2(g) \qquad (6)$$
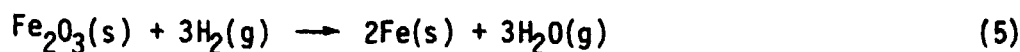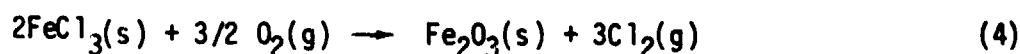
The by-products CO or H_2O can be used to recover oxygen and hydrogen.

(b) Ti extraction: A mixture of Ca and TiO_2 powders is pelletized and heated at 925-950°C.

$$TiO_2(s) + 2Ca(s) \longrightarrow Ti(s) + 2CaO(g) \qquad (7)$$

The Ti produced can be recovered by preferentially leaching CaO with a weak acid wash solution.

c. Other Chemical Processes

R. Waldron (Rockwell Int.), J. Arnold and D. Criswell (California Space Inst.) (Ref. 82) are currently studying chemical processes by acid leaching (HF) and by other processes that depend greatly on water and hydrogen availability in space.

While chemical processes are feasible, they exhibit the following limitations:

o   Different processes have to be designed for each metal.

o   Several steps are needed in order to extract one metal at a time.

o   Oxygen is not a by-product.

o   Require extensive import of consumables from earth (chlorine, fluorine, acids, alkalies, carbon, etc.).

o   Potentially hazardous to manned operations.

o   Difficult to automate.

2.   Electrolysis

The extraction of metals by electrolysis has the advantage of being simple, and that oxygen can be recovered as a by-product. Although the extraction of individual metals is possible, the recovery of a mixture of metals is much easier.

Two types of electrolysis processes will be discussed in this report, the electrolysis of molten silicates and minerals to recover the metals and oxygen, and solid state electrolysis of mixtures of CO_2, CO and H_2O gases evolving from vaporization of carbonaceous chondrites or other chemical processes.

a.   Electrolysis of Molten Silicates

Electrolysis of silicate melts from moon and asteroid materials has to be performed at elevated temperatures, so that the conductivity is high enough for practical production rates. The upper temperature limit for electrolysis of any material will be dependent on the vapor pressure of that material. High vapor pressure may be economically unacceptable because of the loss of materials being vaporized. Data on the conductivity and vapor pressure, as a function of temperature, are available for some pure minerals (Ref. 83-86); additional data will be determined experimentally as required.

The metals which can be obtained by direct electrolysis are magnesium, iron and titanium. Aluminum and silicon can be reduced by secondary reactions. Oxygen is formed at the anode. The electrowinning of oxygen from silicate rocks has been investigated in a NASA sponsored project at the Bureau of Mines by D.G. Kesterke (Ref. 87). Among the minerals that were

electrolyzed were basalts and serpentine. The electrolytic vacuum chamber and cell are shown in Figure VI-11. Fluxing materials had to be used to promote fluidity and electrical conductivity of the molten minerals. As an example, the electrolysis of TiO_2 takes place as follows:

$$TiO_2 \longrightarrow Ti + O_2 \qquad \text{(overall reaction)} \qquad (1)$$
$$Ti^{++} + 4e \longrightarrow Ti \qquad \text{(cathode surface)} \qquad (2)$$
$$2\ O^{-2} \longrightarrow O_2 + 4e \qquad \text{(anode surface)} \qquad (3)$$
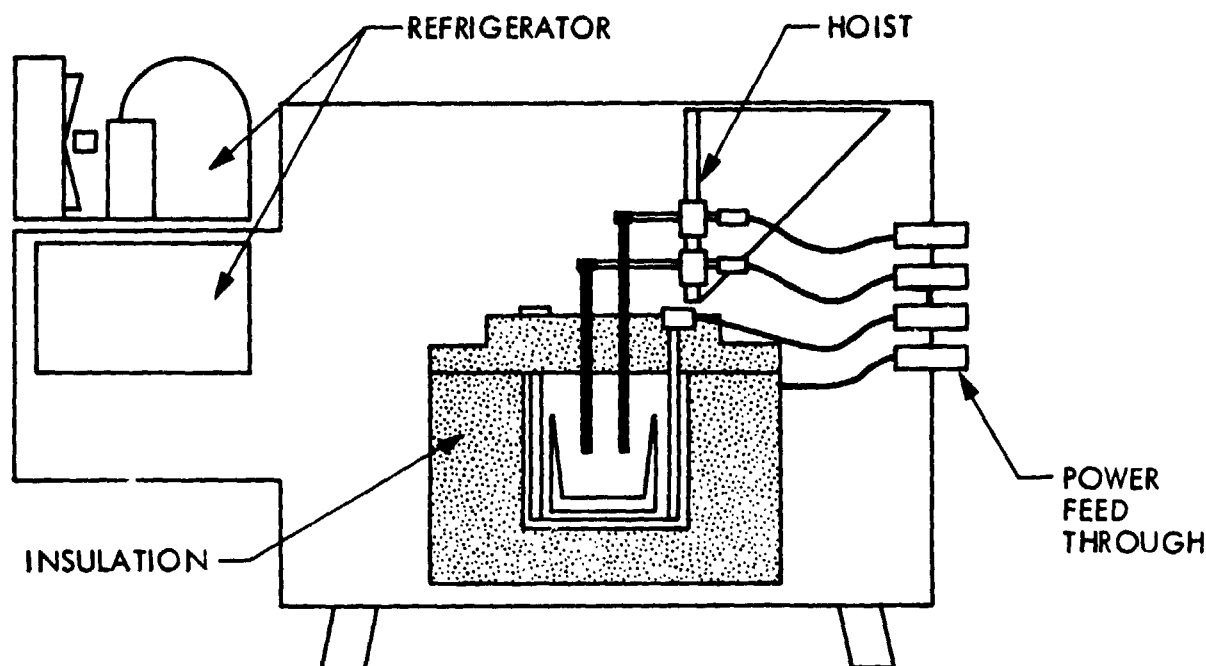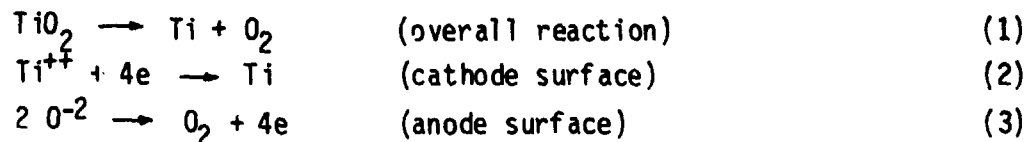
Figure VI-11 Electrolytic Cell in Protective Atmosphere Chamber
(Bureau of Mines, Ref. 87)

In electrolysis of silicate melts it is important to know the nature and the extent of the electrical conductivity. The conductivity is ionic unless a very high content of iron oxide is present, when both ionic and electronic conduction exist. In practical electrolysis it is important to have high current density so that the production rate is kept high.

Electrolysis also demands a knowledge of the decomposition potentials of the species to be deposited. Few directly measured decomposition potentials for silicate melts have been published. Data from linear sweep voltammetry give the so-called halfway potentials which are closely related to decomposition potentials. Halfway potentials for titantium, vanadium,

manganese, iron, cobalt, nickel and copper ions in molten sodium disilicate at $1000^\circ C$ have been determined (Ref. 88). The potentials are in the range from 0.66V to 1.09V. Decomposition potentials can be calculated theoretically from known thermodynamic data. The reverse is also true, i.e., electrochemical experiments can furnish thermodynamic data. Further, the kinetics of electrochemical processes can give the diffusion coefficients of ions present.

There are a number of operational difficulties when electrolyzing at high temperatures. It is necessary to use anodes that are not consumed during the process. Electrolysis on the moon or the asteroids must be self-contained and make use of materials found locally, since the import of materials from the earth must be minimized. The anode can possibly be a gaseous one by using the plasma that can be developed between melt and a conductor situated just above the molten surface. The conductor may have to be cooled to avoid being melted or eroded by sputtering.

The heating of the electrolysis cell may in part be accomplished by the use of concentrated solar radiation to reduce the consumption of electric energy.

Utilization should be made of the existing high vacuum in space, oxygen pressure less than 10^{-14} atmospheres. Oxygen from this cell or any other electrochemical process should be collected and stored in liquid or solid form, preferably in containers situated in moon valleys or canyons which are never exposed to the solar radiation, so that they maintain their very low temperature.

One conceptual configuration of a lunar electrolysis cell for the extraction of metals and oxygen from molten silictes is shown in Figure VI-12. The top section is divided by the charger into an open compartment (at right) for access to direct solar heating, and a closed compartment for the collection of oxygen (at left). Anode consumption is minimized by the use of stabilized zirconia. The metal pool developing in the lower part of the open compartment acts as cathode. To preclude "starving" (loss of conductivity) of the melt, the metal-rich portion in the lower compartment is rotated by an induction coil, imparting a stirring effect upon the entire melt.
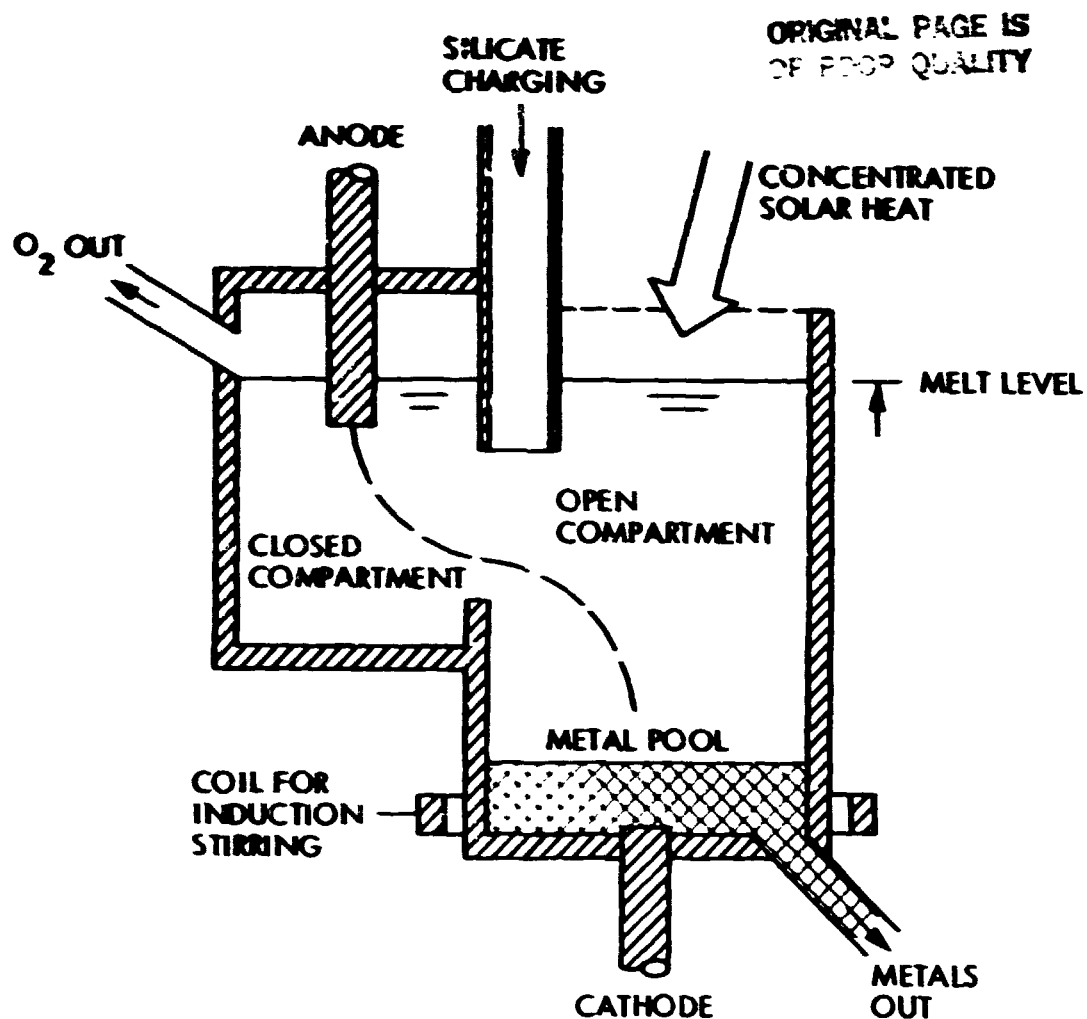
Figure #I-12 Conceptual Electrolysis Cell for Lunar Operations

This concept is essentially a batch process which presents the following problems:

(1) Difficulty, or even impossibility, of maintaining the desired constant electrical conductivity of the melt as it is progressively depleted of metal constituents.

(2) The need for stirring - difficult to implement at the high temperatures - to prevent process decay due to short-circuiting in the melt.

(3) Interrupted production and the need for material transfer and storage which, in turn, requires extensive human activity, highly undesirable and costly in space.

In the attempt to alleviate these problems, recent studies led to the conceptual development of a continuous process for the electrolysis of molten silicates. It provides a continuous gravity-induced flow of the melt through the electrolysis cell and allows effective electrolysis at a constant voltage by variable spacing of the electrodes which offsets the progressive change of electrical conductivity. It further provides for positive separation and collection of the end-products (metals, oxygen and rest-mass), thus permitting direct feeding into product fabrication (single thermal cycle from raw material melting to end-product).

The process requires the presence of an appreciable gravity. In the near-earth regime it applies, therefore, to the moon and the larger asteroids which are the prime bodies of interest for extraterrestrial operations in the foreseeable future.

The process concept is illustrated in Figure VI-13. The digger/conveyor, A, transfers the silicate soil (or comminuted silicate rock), B, into the melting chamber, C, where it is neated by a solar furnace (concentrator). The molten raw material, D, moves into and through the inclined electrolysis cell by gravity. In passing between the anode, E, and cathode, F, both in the form of plates, the molten silicate is electrolytically reduced to liquid metal, G, which flows into the collection chamber, H, and can be directly fed into secondary processing, such as casting, extruding, drawing, etc. The depleted portion of the melt, J, remains separated from the metal due to the large density difference and is removed at point K or likewise fed directly into secondary processing (silicate products). The oxygen, L, precipitating at the anode, E, flows through the tube, M, to the condenser, N, drawn by the difference between the partial pressure of the oxygen in the melt and at the cryogenic condenser plates. The liquid oxygen generated in the condenser is removed at Q for storage.

The flow rate of the molten material through the apparatus is, in the first order, controlled by the gravity-induced hydrostatic pressure, or the elevation difference between the top level in the melting chamber and the collection chamber. The flow rate through the electrolysis cell is further controlled by its inclination. An additional fine-control of the flow rate is achieved by a variable tilt of the entire apparatus, actuated by flow rate sensors and a microprocessor.
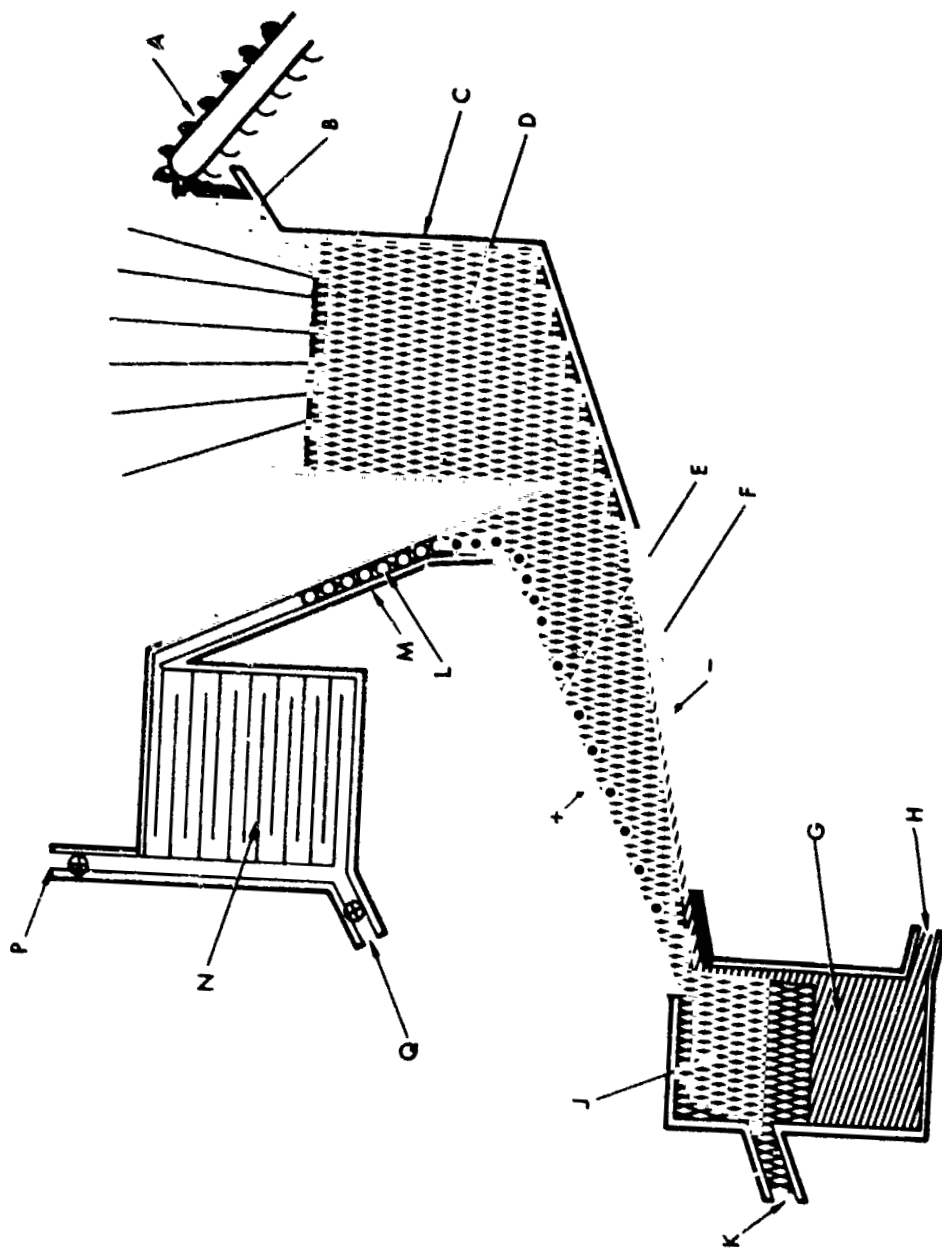
Figure VI-13   Conceptual Apparatus   for Continuous Electrolysis of Molten Silicates

In the flow through the electrolysis cell, the electrical conductivity of the melt decreases as it is depleted of metal ions and cools at increasing distance from the melt chamber. To achieve highest effectiveness of electrolysis, a variable spacing of the electrodes is introduced which has two beneficial effects: (1) the progressive decrease of electrical conductivity along the electrolysis cell is offset by progressively narrowing electrode spacing, thus maintaining uniform electrolytical performance at a constant voltage. (2) Uniform yield of metal and oxygen throughout the length of the cell. These effects are further improved by a variable angle between the electrodes, controlled by microprocessors and sensors monitoring electrical conductivity, temperature, flow rate and other material characteristics of significance for electrolytic performance.

Experimental Investigations

Experiments on the electrolysis of molten silicates focused at the determination of essential material properties which provide a necessary basis for the design of process experiments. They include:

(1) Determination of the electrical conductivities of the three mock minerals.

(2) The determination of the decomposition potentials of species and diffusion coefficients of ions.

(3) Evaluation of refractories, anode and cathode materials, and development use of nonconsumable electrodes.

(4) Determine the practicality of suitable viscosity control, based on available literature data and/or measurements as required.

(5) Study of concentration polarization.

A very simple electrolysis cell was built for the experimental measurement of conductivity as a function of temperature, yield of metal as a function of current density, metal(s) type and concentration as a function of voltage and the consumption rate of electrodes as a function of time and current density. A sketch of the cell is given in Figure VI-14.

MO ANODE

CERAMIC
SPACER

Figure VI-14 Experimental
Electrolysis Cell

X-SECTION
COIL

MOLTEN
MINERAL

MO CATHODE
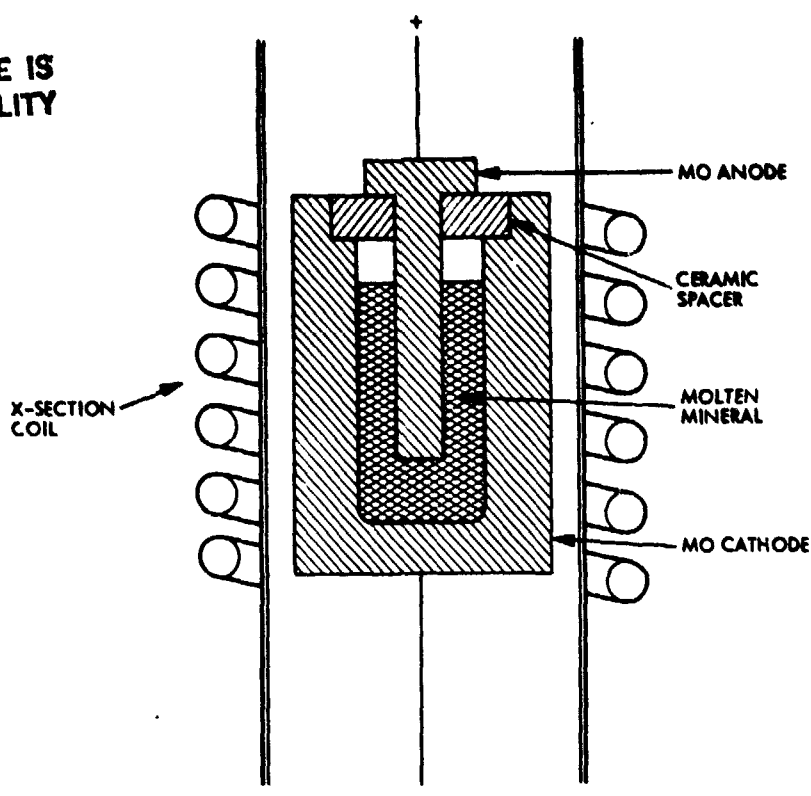
The cell was placed in a quartz tube in the coil of a 50 kW, 450 khz
induction unit. An argon flow of 2 liters per minute was maintained to
prevent coating of the optical window by mineral vapors (temperature
measurements were made with an optical pyrometer). Conductivity measurements
were made, as a function of temperature, for two "mock" extraterrestrial
materials, lunar basalt and lunar anorthosite. The conductivity was measured
by applying two cycles of 60 hz at six volts across the cell. The voltage and
current data bits were placed in storage at the rate of 4×10^5 bits per
second and then plotted out on an x-y recorder for analysis. The initial
conductivity values for the two mock materials are plotted as a function of
temperature in _Figure VI-15_. They range from zero at the mineral melting
point to values appropriate for effective electrolysis in the order of 0.05
and 0.5 ohm cm^{-1} at 00°C. Electrolysis will necessarily have to be
performed at higher temperatures to obtain an efficient yield. This will
cause a more severe corrosion of electrodes and containment materials.

In FY'82 more extensive conductivity measurements will be made at higher
temperatures and on other minerals. The metal types produced, metal yields
and the corrosion rate of containment and electrode materials will be
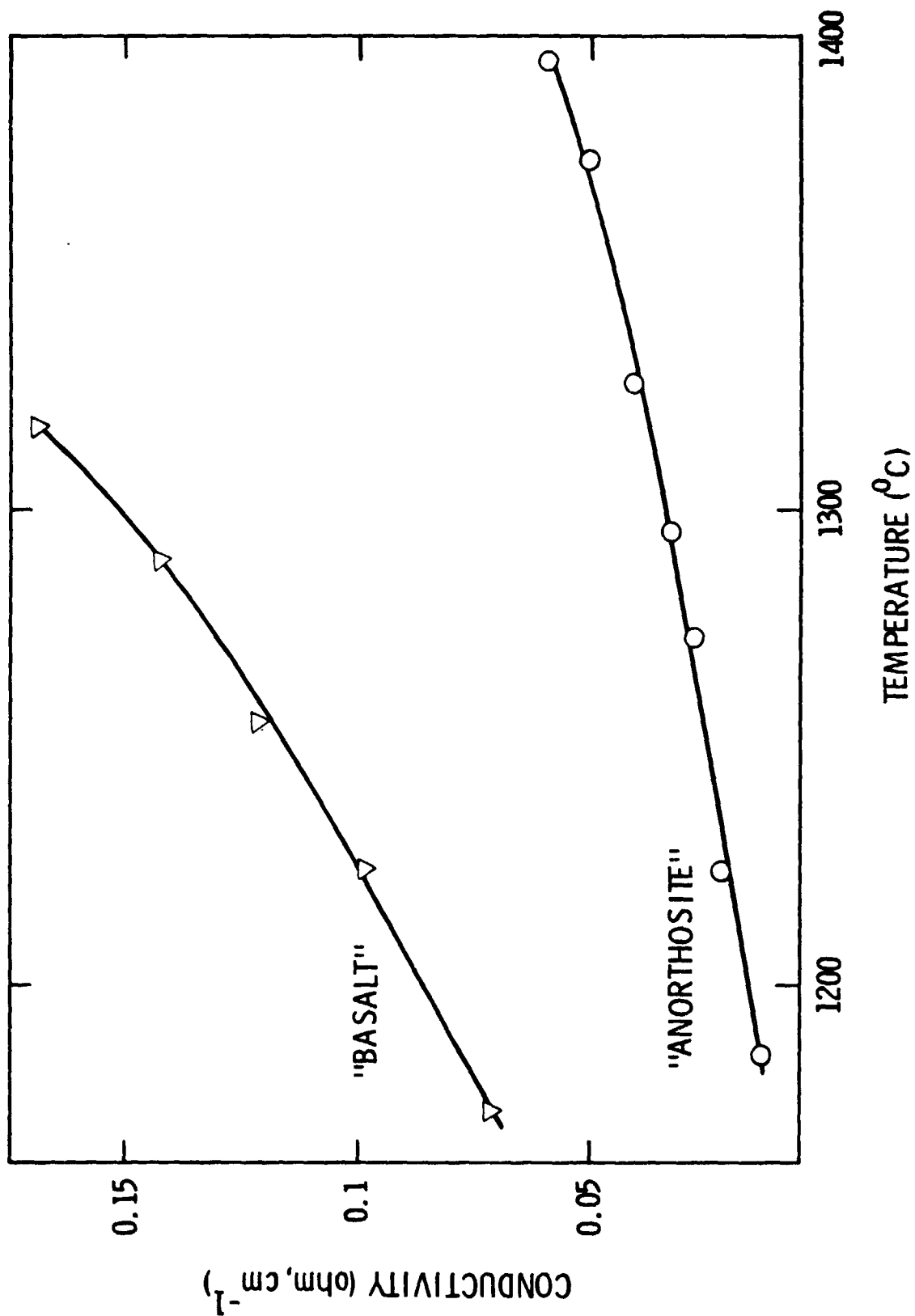
Figure VI-15 Conductivity vs. Temperature for Mock Lunar Materials

determined in the described cell. A more sophisticated cell will be designed for the determination of species decomposition potentials, ion diffusion coefficients and concentration potentials.

b. Solid State Electrolysis

If carbochlorination or other carbon-based reduction processes, or vaporization of carbonaceous chondrites, were performed to produce metals in space, the evolving gases such as CO_2 and H_2O can be electrolyzed to recover oxygen.

INSULATING BOARD
MgO INSULATION
FURNACE
CO_2 OUTLET
O_2 OUTLET
ANODE WIRE (Pt)
CO_2 INLET
CATHODE WIRE
THERMOCOUPLE
COOLANT WATER
2.5 cm
CELL
MUFFLE TUBE
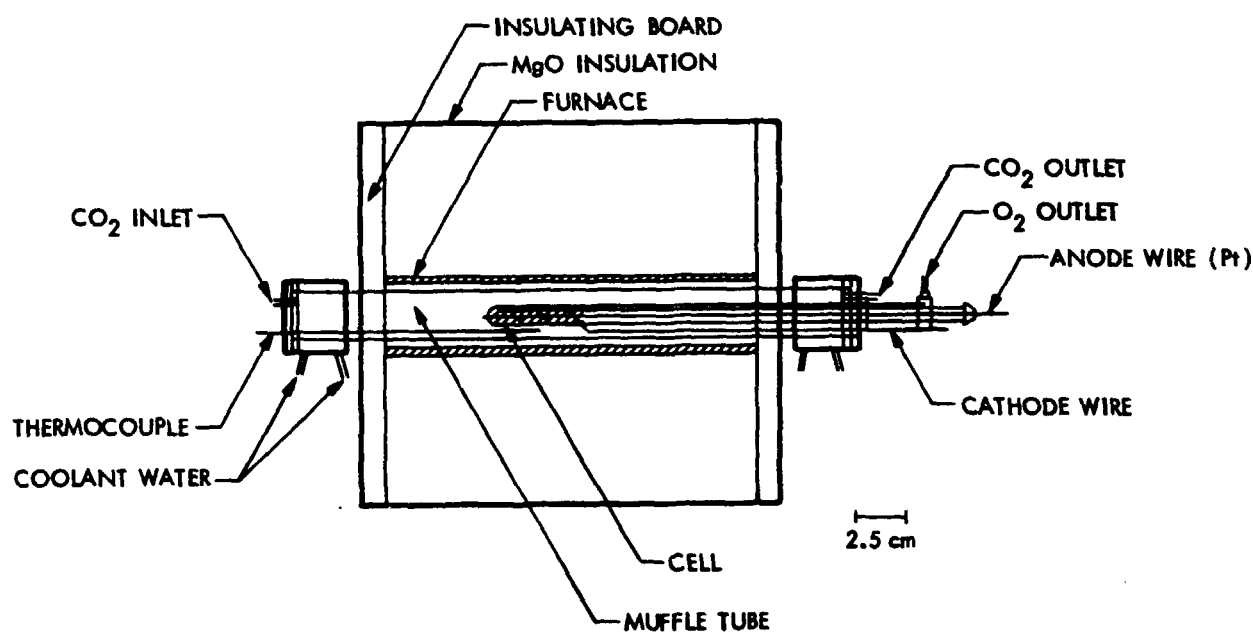
Figure VI-16 Solid-State Electrolysis Apparatus (from Ref. 89)

The recovery of O_2 by solid state electrolysis of a gas mixture containing CO_2, CO and H_2O using a solid electrolyte cell and platinum electrodes (Ref. 86) is being studied. The electrolyte cell is shown in Figure VI-16. During the passage of current through the cell, oxide ions flow through the ceramic due to anion deficiencies in its structure. The reactions are as follows:

At the cathode

$$2CO_2(g) + 4e- \longrightarrow 2CO(g) + 2O^{2-}$$
$$2H_2(g) + 4e- \longrightarrow 2H_2(g) + 2O^{2-}$$

At the anode

$$2O^{2-} \longrightarrow O_2(g) + 4e-$$

The net reactions are:

$$2CO_2(g) \longrightarrow 2C \quad _j) + O_2$$
$$2H_2O(g) \longrightarrow 2H_{2\backslash y)} + O_2(g)$$

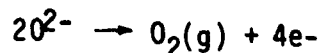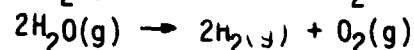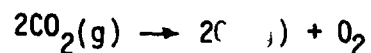The solid electrolyte can be calcia-stabilized zirconia, yttria stabilized thoria, or ceria doped with yttria. A unique feature of this technique is that the continuous electrolysis of CO_2 or H_2O over a mixture of $CO-CO_2$ and H_2O is possible. However, it is not recommended to pursue solid-state electrolysis at the present time because of limited potential applicability (need of consumables).

The electrolysis (or electrowinning) processes are very promising and can be well addapted to space applications. Potential problems are: different process conditions have to be designed for each metal, if metal separation is desired. The present state-of-the-art electrolysis processes require supply of consumable electrodes from earth. However, techniques to minimize this requirement are presently under investigation.

3. Vapor-Phase Reduction

Most of the processes presented in this section, designated generically as "vapor phase reduction", introduce a new concept of extractive metallurgy designed specifically for space operations and thus have no terrestrial counterpart. These processes are based primarily on the availability of an inexhaustible high vacuum and are, therefore, practical on a commercial scale only in space. The method of "selective ionization" (VI-B-3-d) is loosely based on current plasma processing technology; however, it eliminates the addition of reduction agents. All these methods lend themselves to the extraction of individual metals as well as oxygen from metal oxides. By proper vapor deposition techniques they even have the potential of generating metal or metal-alloy shapes in one single processing cycle.

In principle, vapor-phase processes consist of the vaporization of the raw material oxides in fine particle form, dissociation of the vapor and extraction of the individual metal species as well as oxygen from the dissociated gas by various recovery techniques.

The most straightforward method of recovery of metals and oxygen from the dissociated vapor is fractional distillation (Process a). Ionization of the gas species during vaporization permits recovery by deposition on charged plates (Process b) or by electromagnetic techniques (Process c), or by selective ionization (Process d). In all cases, gas flow and pressures in the processing column are maintained and controlled by the pressure differe :ial between the point of raw material vaporization and a variable-size orifice at the end of the column which opens to the environmental vacuum of 10^{-14} torr.

a. Vaporization/Fractional Distillation

This process consists of (1) vaporization of the raw material oxides and dissociation of the gas, resulting in gaseous metal and oxygen molecules; (2) recovery of individual metals and oxygen by fractional distillation at discreet temperatures. This process is attractive with regard to simplicity and energy consumption.

A conceptual configuration of the apparatus is shown in Figure VI-17. This sketch, as well as the two following, should be regarded as process illustration in principle, rather than equipment design. The raw material size (<100 μm particles) is dispensed from the container "A" through an orifice "B" in a fine particle stream which is vaporized in an induction-heated conical susceptor ("C"). Under the pressure gradient from 10^{-2} torr at A-B-C to 10^{-4} torr at the end of the column "G" and 10^{-14} torr beyond the control orifice, the gas moves at high velocity through Sections D-E-F. In the distillation units "E" the gas is forced through a labyrinth of plates cooled to the respective condensation temperature, resulting in the recovery of individual metals by distillation. Oxygen may be recovered in the same way in Section "F", or blown overboard through orifice "G" together with metal losses and trace elements.
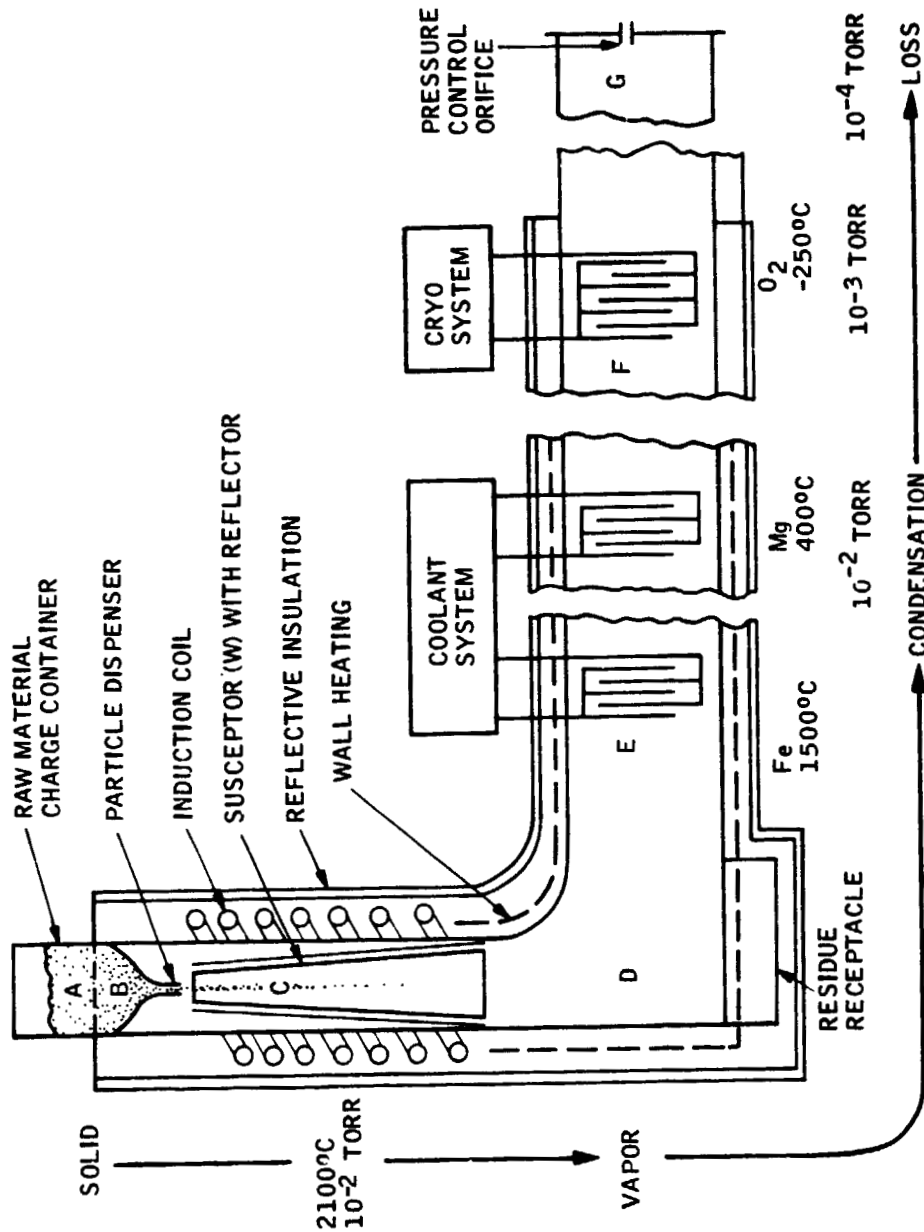
Figure VI-17 Vapor-Phase Reduction: Recovery of Metals and Oxygen
by Fractional Distillation. (Conceptual)

Considering losses in the distillation process, the theoretical metal yield is approximately 44.5% (of soil weight), and the required energy 34,000 kWh/ton of soil which has to be supplied regardless of the recovered species.

Since this process depends on the volatility of the different species, it is important to determine their vapor pressures. Under this investigation, both theoretical and experimental studies are being performed.

A thermodynamic approach was used to determine stability/instability data for the oxides and silicates, vapor pressures and relative volatility of the various species. The basic Hertz-Knudsen-Langmuir equation was used:

$$\text{Flux } J_i = \frac{P_i}{(2\pi M_i RT)^{\frac{1}{2}}} \text{ moles/area,time}$$

The calculated vapor pressures of the different species were very low and were of the same order of magnitude in several cases. As an example, in the $(MgSiO_3)$ system, it was 10^{-5} torr for Mg, and 4×10^{-6} torr for Si. The prime problem of this process is the expected recombination during the distillation phase. This may be prevented by the use of reduction agents, either in the form of fine powders, such as Al or carbon, or in the form of gases, such as hydrogen. The import of these reduction agents, not readily available in space, would defeat the purpose of this process. However, hydrogen may be obtained from permafrost on the moon (which is still speculative, see Section III-C), and carbon from carbonaceous chondrites. A modified recovery concept is presently under study which promises to eliminate or, at least, minimize the need for reduction agents.

b.   Ionization / Electrostatic Vapor Deposition

In this process concept, the oxides are vaporized, as in the foregoing process, then ionized, subsequently or as part of the vaporization process, resulting in positive metal ions and negative oxygen ions. During the flow of the ionized gas through sets of charged plates, the metal ions are deposited at the negative and the oxygen at the positive plates. The metals may either be deposited as a mixture or individually by process refinement.

In the conceptual illustration, <u>Figure VI-18</u> the oxides (soil) are
dispensed on a cooled turntable at point "B" and rotated to point "C" where
they are vaporized and ionized by an electron beam or a laser beam. To
minimize the gas loss through the necessary opening for the electron beam, the
pressure is gradually reduced from the 10^{-2} torr in the vaporization chamber
to the ambient 10^{-14} torr by a sequence of chambers ("pressure baffles").
The positive metal ions are recovered by passage of the gas flow through the
negatively charged plates "E" and the negative oxygen ions at the positive
plates "F". Labyrinth arrangement of the plates enhances turbulence for a
high degree of ion-plate contact, and consequently, high recovery yield.

Theoretically, this process requires an energy of approximately 62,000
kWh/ton of metals. If the oxygen is also recovered, this figure reduces to
44,000 kWh/ton (metals and oxygen).

The product obtained at the negatively charged plates is a metal alloy
whose composition is determined by the relative metal content of the
feedstock. It is conceiveable that individual metals may be extracted by
tuning of the plate charge to the mass/charge ratio of specific metal ions.

c.   Ionization / Electromagnetic Recovery

The individual species of an ionized gas stream may also be
recovered by deflection of their path in an electromagnetic field similar to
the principle of the mass spectrograph. In this process concept, the oxide is
vaporized and the species ionized by a high-intensity electron beam. In the
passage through the field of a strong electromagnet, the ions are deflected
toward the column wall at distinctly curved paths in accordance with their
mass/charge ratios. All metal ions (positive) as well as some potentially
positive oxygen ions move to one side and hit cooled recovery ring sections at
distinct distances from the magnetic field, while the predominantly negative
oxygen ions move to the opposite side.

The illustration of this process in <u>Figure VI-19</u> shows only the recovery
section of the processing column as the vaporization/ionization section may be
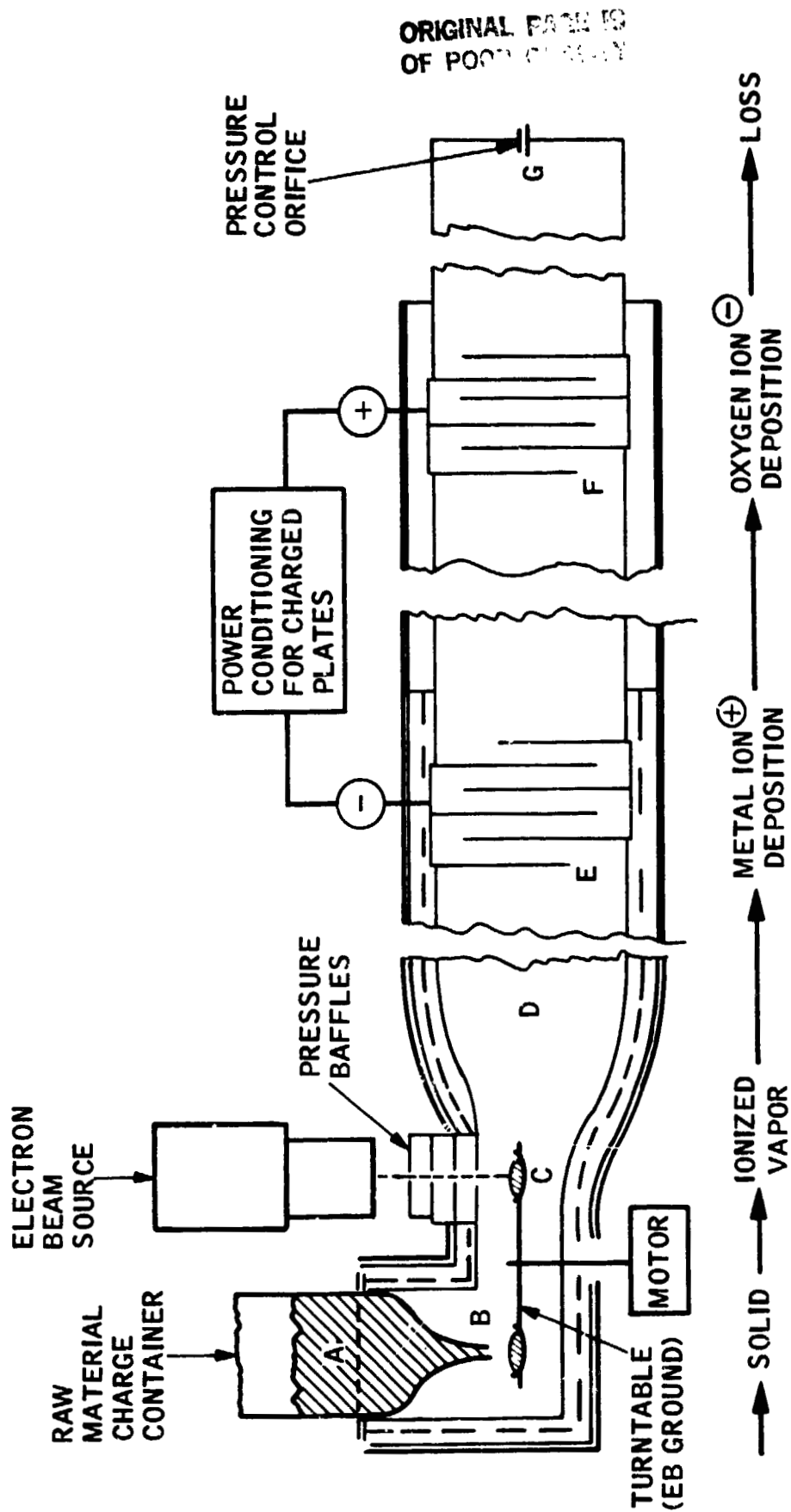identical to Process b (Figure VI-8). As the ionized gases pass through the

Figure VI-18 Vapor-Phase Reduction: Electrostatic Recovery of Metals and Oxygen. (Conceptual)
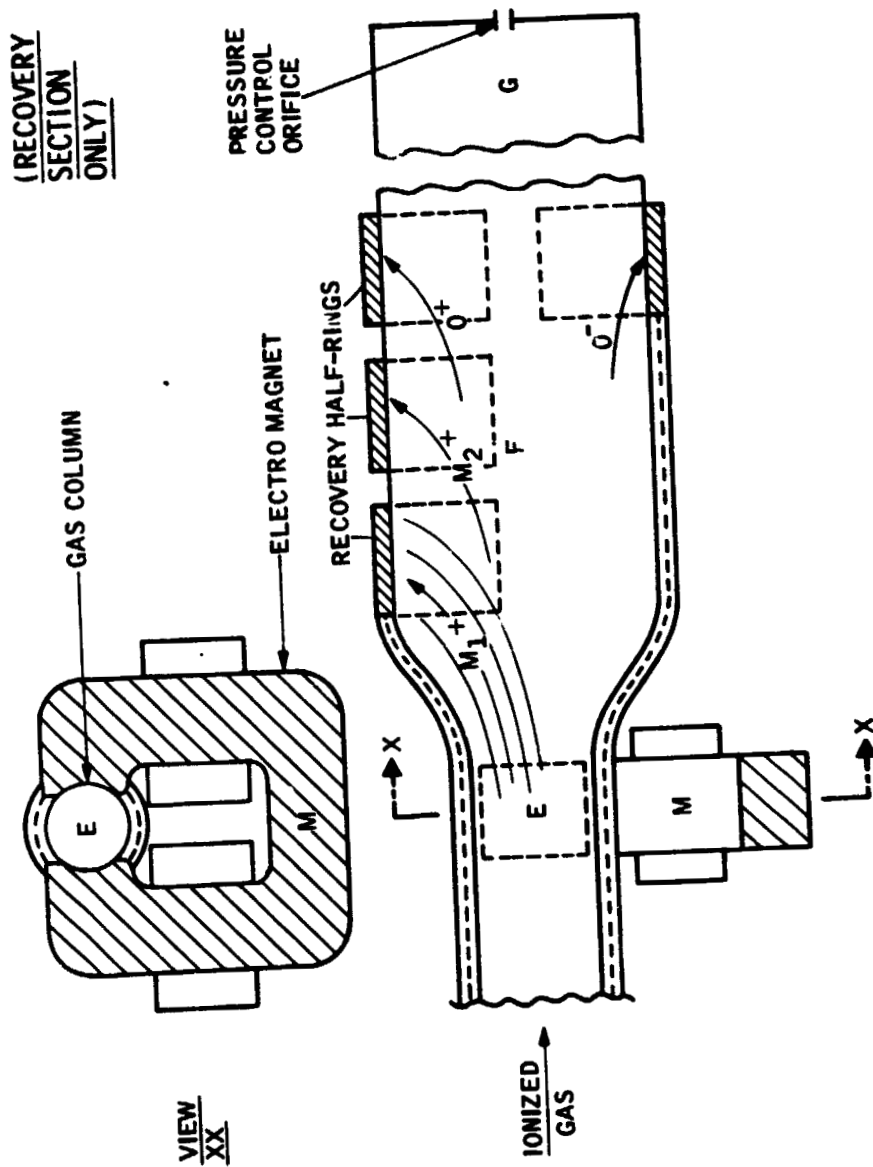
Figure VI-19 Vapor-Phase Reduction: Electromagnetic Recovery of
Metals and Oxygen. (Conceptual)

field "E" of the electromagnet "M", the path of individual species is deflected toward the cooled recovery half-rings "F". The path curvature is defined by the mass/charge ratio of individual species and the gas flow rate, while the path direction is defined by the charge sign (+ or -).

While the recovery yield is expected to be essentially the same as in the previous process, the theoretically required energy is substantially higher (72,000 kWh/ton of metals and 96,00. kWh/ton of oxygen).

This process produces individual metals of high purity. In both, processes b and c, it is further conceiveable that by appropriate configuration and coating of the deposition elements (negative electrcdes in b and targets in c) shaped metal products, such as strips, channels or tubings, can be obtained directly from the vapor phase.

d.   Selective Ionization

The conceptual studies of processes b and c, above, provided an information base for process refinement. They indicated that high temperature ionization (plasma techniques) are most suitable for space applications, since they are versatile sources of energy which is required for the decomposition of metal oxides or silicates at very high temperatures. This conclusion led to a new concept of metal/oxygen separation in a high temperature plasma, designated as "Selective Ionization".

This process takes advantage of the greater percentage of ionization of metals over oxygen within a certain temperature range of a thermal plasma, see Figure VI-10 (Ref. 90-93). This figure shcws that at a discrete temperature metals of interest, such as Al, Mg, Ni, Fe are highly ionized, while gases such as O, N or Ar are still essentially neutral. This permits an effective separation of metals and oxygen. Furthermore, the careful control of process temperatures may allow the attainment of pure etals and alloys of varying composition.
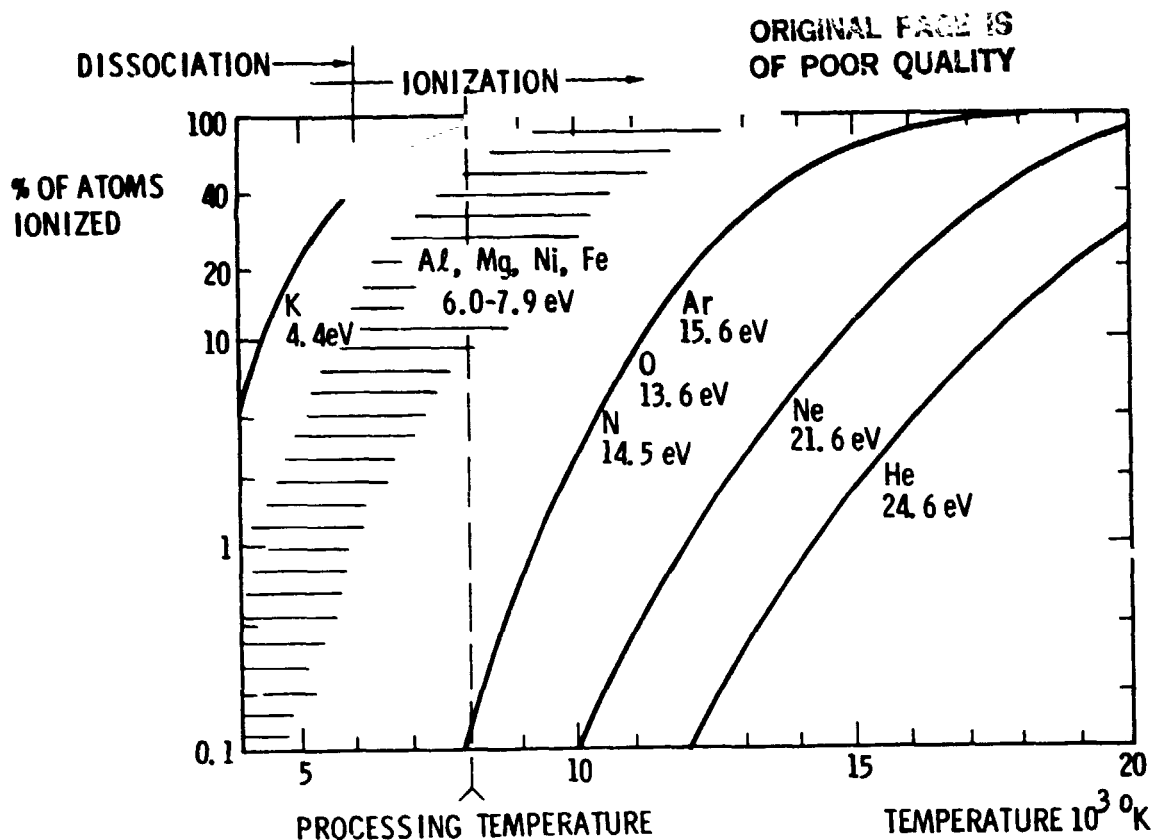
Figure VI-20   Degree of Ionization of Various Elements
in a Thermal Plasma

A conceptual illustration of this process is shown in <u>Figure VI-21</u>. The
powered raw material (A) is fed through an orifice, where it is vaporized,
atomized and ionized upon entering the fireball (B) of an induction-coupled
plasma.   At a process temperature of approximately 8000°K the plasma is
acted upon by either an electrostatic (C) or magnetic field, diverting only
the charged metal ions, while the essentially neutral oxygen remains
unaffected and continues on the downstream path.   (At 8000°K, preliminary
calculations show that 40-80% of the metals are ionized while only 0.1% of the
oxygen is charged.)*   Oxygen may be recovered by adiabatic expansion and
collected as a liquid or blown overboard together with metal losses and other
solids.

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*   Other work (T. Yoshida et al, Ref. 94) suggests that better than a 90%
      yield of 95% pure iron is achieved at 10,000°K in an iron-oxygen system.
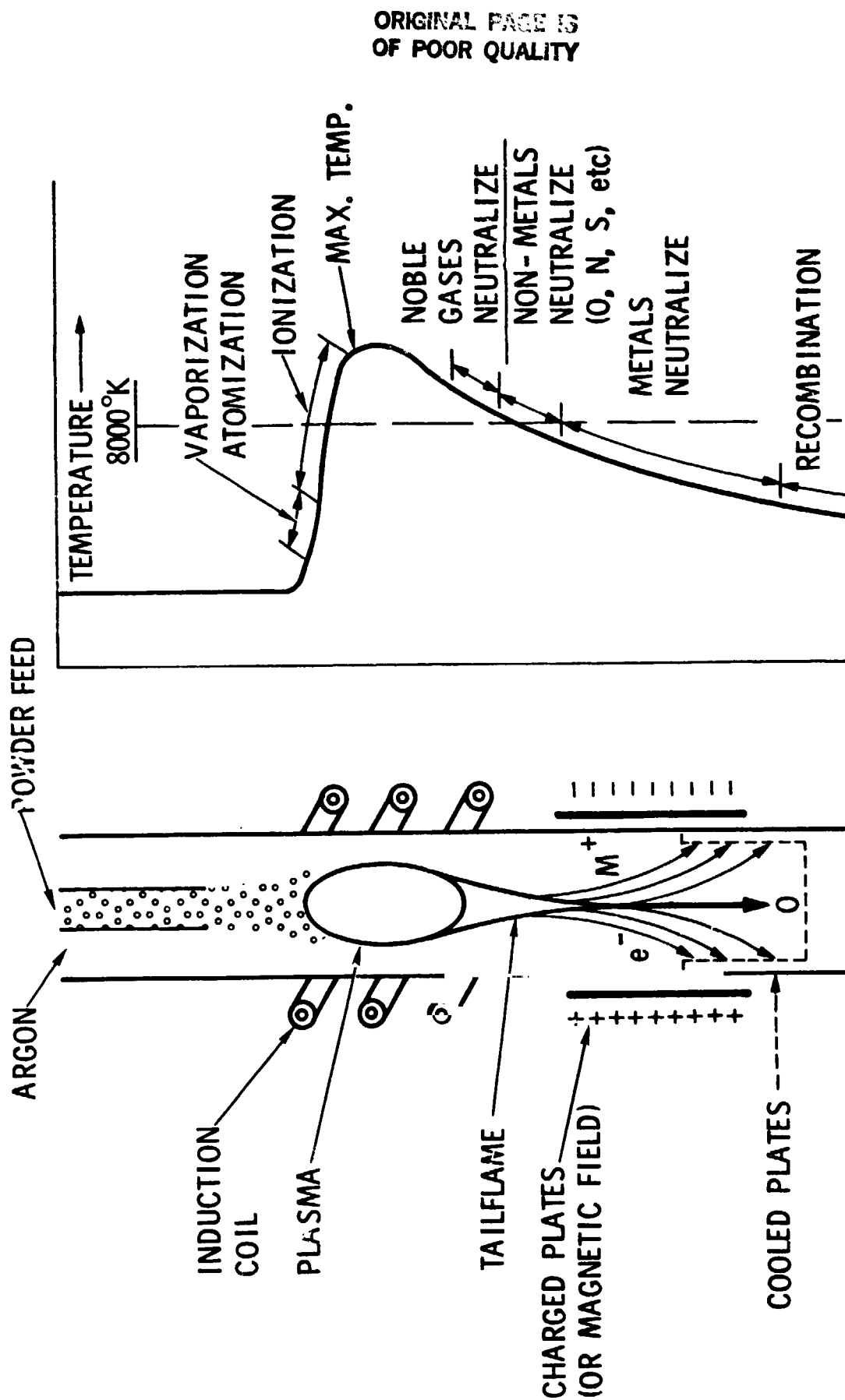
Figure VI-21 Selective Ionization: Conceptual Apparatus Configuration and Temperature Profile

The assessment of the theoretical and practical power requirements in kWh/ton is presently in progress. Preliminary calculations indicate lower power requirements than for the previously described processes b and c.

Determination of the gas ionization potentials of the different species, at different temperatures is underway. The degree of ionization of individual species (% ionized vs. temperature) is being calculated using the Saha equation:

For single ionization:

$$\frac{\alpha^2}{1 - \alpha^2} = \left(\frac{2\pi \cdot m_e}{h^2}\right)^{3/2} \left(\frac{kT}{p}\right)^{5/2} \frac{2 Z_1}{Z_0} e^{-u_i/kT}$$

For multiple ionization:

$$\frac{n_{i+1} \cdot n_e}{n_i} = \frac{2 (2\pi m_e kT)^{3/2}}{h^3} \frac{Z_{i+1}}{Z_i} e^{-(u_{i+1} - u_i)/kT}$$

n_{i+1} = number of excited atoms in i+1 state.
n_e = number of electrons.

The feasible techniques for ionization of gases were studied, namely: by electron beam gun, arc-discharge, RF and laser techniques. The electron beam technique was discarded for the following reasons: the ionized gases must be immediately recovered on charged plates very close to the beam action area which is very small and the lifetime of the ionized state is too short to prevent recombination. In the arc-discharge plasma, an inert gas may be necessary, such as H_2 to avoid corrosion problems. Both RF (induction) and laser techniques appear attractive. The RF technique was ultimately selected for the following reasons:

1. It appears most effective for the generation of a thermal plasma.

2. It exhibits the low power requirements, particularly at higher frequencies. The 2-10 Mc band appears to be most efficient.

3. It was almost exclusively used in applicable work reported in the literature.

e.   Theoretical and Experimental Studies of Processes B-3

The objective of the theoretical and experimental studies is to evaluate the four discussed processes and define the most technically and economically feasible process for extraction, recovery and shaping of the metals in space. Some of the processes will be carried out on earth in ultrahigh vacuum, i.e., various vapor deposition techniques. The produced plasma and materials will be characterized. The following theoretical and experimental work is either in progress or in preparation:

(1) Vaporization of the silicates and/or oxides:

Theoretical Studies:

o   Establishing accurate data for the stability of the metal silicates and/or oxides, by thermochemical evaluation (temperature, free energy, energy required).

o   Determination of the dissociation/recombination of silicates and/or oxides, into the corresponding constituent metals and gases, by thermochemical evaluation (temperature, pressure, free energy and energy required).

o   Determination of the vaporization mechanisms involved in the dissociation reactions of the silicates and/or oxides, vapor pressures of the various species, the relative volatility of the species and the maximum achievable rates of vaporization.

o   Potential elimination of reducing agents by rapid quenching techniques.

Experimental Studies:

o   Evaluation of the fractional distillation process using initially aluminum powder as a reducing agent.

o   Potential elimination of reducing agents by appropriate techniques, such as rapid quenching.

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o   The feasibility of the fractional distillation technique will
     be determined by comparing the experimental results and
     theoretical data.

(2) Ionization/Plasma Techniques, of the metal silicates and/or oxides:

Theoretical Studies:

o   Determination of the density of electrons produced in the
     ionized state as a function of gas temperature, of the
     different species. Also, determination of gas pressures, gas
     ionization potential and energy required, at different
     temperatures.

o   Evaluation of the plasma flow and ion separation by
     electrostatic and electromagnetic fields actions.

o   Determination of the rate of ionization and/or recombination of
     ions and electrons in the three proposed processes (b), (c) and
     (d).

o   Assessment of the expected yield (metals, oxygen) and the power
     requirements in terms of kWh/ton.

o   Conceptual facility design studies.

Experimental Studies:

     Of the four vapor-phase techniques described above, three,
fractional distillation (a), electrostatic (b) and electromagnetic (c)
separations depend on low pressures or reducing agents to avoid possible
recombination to form metal oxides. Low pressures dictate low mass
throughputs and reduction agents must be shipped from earth; neither
alternative is desirable. Selective ionization is not dependent on either of
these alternatives because the metals are removed at a temperature above which
stable oxides form. Thus, selective ionization will be emphasized in
experimental investigations during FY'82. The experimental plan which is
designed for a stepwise build-up of capabilities from a pure gas plasma to the
exclusive use of metal oxides (without carrier gas) is as follows:

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MATERIAL		OBJECTIVES
(1) Argon (only)	o	Techniques to start ionization
	o	Gas flow characteristics for
		plasma containment, sustainment.
(2) Ar + Metal Particles	o	Plasma Diagnostics
	o	Cold plate recovery of metals
(3) Ar + Oxide Particles	o	Electrostatic recovery of metals
	o	Degree of gas (Ar, O) neutralization
(4) Oxide Particles (only)	o	Plasma Initiation (e.g., Sodium)
	o	Pressure/temperature optimization
	o	Refinement of metal recovery system
	o	Recovery of oxygen by decompression
(5) Process Demonstration	o	Implementation of Results of (4).

Exploratory experiments for the development of an argon plasma "torch"
were conducted with a 450 kHz induction unit, as this was the most readily
available frequency. Though these experiments proved the feasibility of using
this low frequency, a frequency in the megacycle range is desirable. A
megacycle induction generator ignites and maintains a plasma more easily and
more efficiently, important factors for autonomous operation in space. The
procurement of a megacycle induction unit is planned for FY'83, pending
determination of the most effective Mc-band permissible under FCC regulations.

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GLOSSARY OF TERMS

The following list of adopted terms is designed to define their specific meaning as applied to extraterrestrial processing. It will be augmented and refined as the program progresses.

Mining and Conditioning	Comprises all operations to convert the raw material, from its dormant in-situ state as an extraterrestrial material, into the condition required for processing (feedstock).
Acquisition	Self-explanatory (equivalent of terrestrial mining).
Comminution	Break-up of rock or breccia into smaller pieces or a specific particle size.
Beneficiation	Separation of useful components of soil or comminuted raw material from less useful components. Results in enriched feedstock.
Sizing	Separation of soil or comminuted material by particle size, in most cases to a desired maximum size.
Feedstock	Raw material ready for processing - end product of Mining and Conditioning.
Dressing	Addition of specific materials to the feedstock to enhance processing effectiveness.
Processing	Comprises all operations to convert feedstock into products.
Primary Processing	Produces desired materials in bulk or in the form of basic shapes.

Primary Product	Result of primary processing.
End Product	Result of total processing sequence (primary + secondary).
Secondary Processing	Production of finished shapes or complete system assemblies.
Extraction	Recovery of desirable species, such as metals, water, oxygen, etc. from feedstock.
Refinement	Improvement of material quality with regard to composition or properties.
Fabrication	Manufacturing operations used in the transition from basic shape to final required product shape, dimensions or configuration.
Post-Treatment	Operations to modify or improve product properties without affecting dimensional characteristics.
Assembly	Joining of like or unlike products into a functional subsystem.
Installation	Integration of subsystems into a system.
Imports	Supplemental materials or chemicals required in processing and supplied from earth.
Throughput	Mass of material input into beneficiation or processing per unit of time for a given facility.
Output	Mass of useful material in mass/time obtained in beneficiation or processing.
Output Factor	Ratio of output to throughput.
Net Output	Output minus imports (mass/time).
Energy Requirements	kWh/ton
Power Requirements	In kW for a specific throughput rate.
Electric	(Solar-electric)
Direct Solar	(Solar furnace)

Appendix B

SOURCES OF ASTEROID INFORMATION

Data on the composition and structure of asteroids are inferred from three distinct sources: laboratory analysis of meteorites, meteor (the streak cf a "falling star") physics, and spectral studies of asteroids in space. Of these, the evaluation of meteors is most subjective, due to the difficulty of understanding and interpreting the physical processes that lead to the observed spectra. The spectral classification (C, S, M and U) categories bear only loose connections to their mineral/compositional counterparts due to the spectral effects of particle size and surface condition (Ref. 1). The laboratory analysis of meteorites is precise, but it may not be representative of the asteroid belt as a whole due to the sampling effects discussed below. However, what we sample as meteorites (except for the atmosphere breakup and field recognition effects) should be representative of near earth objects (earth crossers and cometary debris). Since we are planning to place emphasis on near earth resources, it is convenient that we have meteorite samples which presumably represent the bodies that we expect to encounter in our search for raw materials in the near earth regime.

1. Effects of Meteorite Sampling

If a mass of material enters a planet's atmosphere it does one of three things: it may disintegrate in the atmosphere, it may ablatively cool and slow down until it is at terminal velocity, or it may impact the earth near "cosmic" velocity. At terminal velocity (100-200 m/s) it no longer glows, but falls 'like a rock' and produces a hole a few feet deep on impact. From this type of impact we can collect meteorites. If it impacts at cosmic

velocity, its kinetic energy is converted to heat almost instantaneously resulting in an explosion-like event. This produces craters like the Barringer crater in Arizona. If it disintegrates in the atmosphere it causes a blast but no crater (example: the Tunguska explosion). What we can sample as meteorites is only what is not completely destroyed during encounter with the atmosphere or the surface (Ref. 2).

About half of the falls break up in the atmosphere, and these multiple falls are more common for weak types than for strong types. Other than the mechanical breakup, the atmospheric passage does not affect the interior of the meteorite. Meteorites are cool inside and one has even been said to show frost on an inside surface cracked open on impact. Heat of ablation penetrates only one cm in nickel iron meteorites, and less in other types (Ref. 3). The entire entry-impact event is on the average ten seconds long. Thus entry does not affect the material itself, but it controls what materials are recovered.

Meteorites in museums and collections are probably not representative samples of all the asteroidal material because filtering of various types occurs during their transport to earth and into collections. While some meteorites are proportionately more common in collections than they would be in space, there are others which are scarce relative to their abundance in space. The filtering mechanisms are as follows:

o   Iron meteorites and stony irons are easy to find because they don't look like ordinary earth rocks, are much denser and are therefore picked up more often as a "find". In contrast, the stony meteorites (chondrites and achondrites) are not adequately represented in the finds.

o   Carbonaceous chondrites are also underrepresented because they are more friable and tend to break up in the upper atmosphere, often not even reaching the ground. They also decompose relatively quickly on exposure to surface conditions.

By examining the "fall statistics" for various meteor and meteorite types, we can estimate what types are in the greatest abundance in near Earth space. Those which have orbital parameters which take them close to the

earth, and are thus resource candidates, are the most likely to be swept up by the Earth. The density of asteroidal material may play a part in determining the probability of earth encounter, since a high density object is less likely to be thrown off course in a collision.

Ordinary chondrites are the most common meteorites observed to fall. Nickel-irons are much scarcer. The vast majority of material encountering the earth is burned up in entry. The spectra of the meteors is most often indicative of carbonaceous material, thus suggesting that most material entering the atmosphere is carbonaceous in nature (Ref. 4). The reason for so few carbonaceous specimens in collections is presumably due to the sampling effects mentioned above. Since the ordinary chondrites are the most common rock type to make it through the atmosphere, they are presumably the next most common type to be found in near Earth space. Nickel-irons are the least common. In order to determine quantitatively how common each type is relative to one another, the quantitative behavior of the sampling effects would have to be determined.

2. Composition and Structure of Meteorites

For the purpose of this study, the meteorites may be classified into four broad groups:

(a) Ordinary Chondrites
(b) Carbonaceous Chondrites
(c) Achondrites
(d) Irons and Stony Irons.

In the following, each of these groups is discussed individually with regard to composition and structure.

a. Ordinary Chondrites

The ordinary chondrites are by far the most common rocks besides carbonaceous chondrites to encounter the Earth. They are stronger than the carbonaceous chondrites and are poor in volatiles. They contain chondrules which are surrounded by a finer crystalline matrix. They are divided into

three groups - H, L, and LL. H refers to high iron content and L refers to low iron. LL refers to low iron, low metal. The H and L types are the most common, with L being slightly more common than H. Their compositions are given in the Table B-1.

Table B-1 Approximate Composition of Ordinary Chondrites

H-Bronzite Chondrites

Mineral	Form in Rock	Description	Weight Percent
Troilite	matrix	FeS	5%
Olivine	chondrules/matrix	$(Mg,Fe)_2SiO_4$	25-40%
Bronzite (a pyroxene)	chondrules/matrix	$(Mg,Fe)_2SiO_3$	20-35%
Ni-Fe	matrix	Ni,Fe	16-21%
Apatite	matrix	$Ca_5(PO_4)_3(F,Cl,OH)$	few%
Merrillite/Whitlockite	aggregates	$Ca_9(Mg,Fe)H(PO_4)_7$	1%
Chromite	matrix	$FeCr_2O_4$	few%
Diopside	chondrules/matrix	$CaMgSi_2O_6$	few%
Oligoclase (a plagioclase)		$(Ca,Na)AlSi_3O_8$	10%

L-Hypersthene Chondrites

Mineral	Form in Rock	Description	Weight Percent
Troilite	matrix	FeS	5%
Olivine	chondrules/matrix	$(Mg,Fe)_2SiO_4$	35-60%
Hypersthene (a pyroxene)	chondrules/matrix	$(Mg,Fe)_2SiO_3$	25-35%
Ni-Fe	matrix	Ni,Fe	8%
Apatite	matrix	$Ca_5(PO_4)_3(F,Cl,OH)$	few%
Merrillite/Whitlockite	aggregates	$Ca_9(Mg,Fe)H(PO_4)_7$	1%
Chromite	matrix	$FeCr_2O_4$	few%
Diopside	chondrules/matrix	$CaMgSi_2O_6$	few%
Oligoclase (a plagioclase)		$NaAlSi_3O_8$ plus a little Ca	10%

b. Carbonaceous Chondrites

Carbonaceous chondrites are relatively friable volatile-rich rocks which generally contain carbon (hence their name, although they are not the only rock types with carbon present). The best known and probably also most common carbonaceous chondrites have been classified as C1, C2 and C3. C1's are primarily fine grained matrix while the high numbers have more chondrules, less volatiles, and a coarser matrix (Ref. 6). The matrices of the C1 chondrites are especially volatile-rich and are only stable below $500^{\circ}K$. Their compositions are given in Table B-2. The data often differ by a few percent, so the listed amounts should be considered approximate (Refs. 2, 3, 5, 6).

Table B-2 Approximate Composition of Carbonaceous Chondrites

Mineral	Form in Rock	Description	Weight Percent
CI			
Serpentine	fine matrix	complex	1%
Chlorite	fine matrix	mixture of Mg,Si,OH,Fe	90%
Montmorillonite	fine matrix	Al,O,Ca and Na	1%
Troilite	fine matrix	FeS	few%
Pentlandite	fine matrix	(Fe,Ni)S	1%
Magnetite	fine matrix	Fe_3O_4	few%
Gypsum	in veins	$CaSO_4 \cdot 2H_2O$	1%
Epsomite	in veins	$MgSO_4 \cdot 7H_2O$	1%
organics & graphite	fine matrix	CHO etc.	few%

Serpentine	fine matrix	complex	1%
Chlorite	fine matrix	mixture of Mg,Si,OH,Fe	50%
Montmorillonite	fine matrix	Al,O,Ca and Na	1%
Troilite	fine matrix	FeS	few%
Pentlandite	fine matrix	(Fe,Ni)S	1%
Magnetite	fine matrix	Fe_3O_4	few%
organics & graphite	fine matrix	CHO etc.	few%
Sulfur	in matrix?	S	1%
Olivine	chondrules	$(Mg,Fe)_2SiO_4$	1%
Enstatite	chondrules	$MgSiO_3$	50%
Ca, Na and Mg Salts		$CaCO_3$, $CaMg(CO_3)_2$, $MgSO_4$ H_2O and others	0-10%

Mineral	Form in Rock	Description	Weight Percent
Plagioclase	chondrules	$NaAlSi_3O_8$-$CaAl_2Si_2O_8$	few%
Ni-Fe metal	fine matrix	Ni,Fe	few%
Spinel	aggregates	$MgAl_2O_4$	1%
Alumina	aggregates	Al_2O_3	few%
Merrillite/ Whitlockite	aggregates	$Ca_9(Mg,Fe)H(PO_4)7$	1%
Troilite	fine matrix	FeS	few%
Magnetite	fine matrix	Fe_3O_4	few%
organics & graphite	fine matrix	CHO etc.	1%
Olivine	chondrules/ matrix	$(Mg,Fe)_2SiO_4$	80%
Enstatite/ Hypersthene	chondrules	$MgSiO_3(Mg,Fe)SiO_3$	5%

c. Achondrites

The basaltic achondrites are the closest analogs to lunar samples in terms of chemistry and petrology. They vary in composition from almost pure pyroxene (diogenites) to a mixture of various minerals found in other meteorites including plagioclase, pyroxene, olivine, Ni-Fe, triolite,

chromite, graphite, schreibersite, oldhamite, diamond, ilmenite and quartz. They contain mostly plagioclase, pyroxene and olivine. Their crystals are interlocking as if formed from a melt and their strengths should be accordingly similar to terrestrial basalts if fracturing from shock effects can be neglected. Their plagioclase content may be the highest among the meteorites (up to 32%) (Ref. 7).

d.   Irons and Stony Irons

Most of the volume of these bodies is composed of nickel-iron metal. Many minerals found in the ordinary chondrites are also found in the Ni-Fe meteorites and make up the remaining 0-70% of the composition. The silicate minerals are segregated from the metal phase since their molten phases are immiscible. In mesosiderites, the materials may be mixed on a scale of millimeters. Iron and stony iron asteroids have the following composition ranges:

       Ni-Fe   Ni,Fe (Ni 4-60%), Fe 40-96%) 30-100%
       Silicates   0-70%

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